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Study on Slurry Flow Characteristics and Diffusion Law of Superfine Cement-Based Composite Grouting Material

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Abstract: The soft surrounding rock of deep roadways is in the state of "micro-fracture and low permeability". In order to solve the problem of grouting reinforcement of micro-fractures surrounding rock in deep roadways, the influence characteristics of auxiliary materials and additives on slurry flow were analyzed, and the composition and proportion of superfine cement-based composite grouting materials were determined: superfine cement accounted for 89.4%, superfine coal ash accounted for 5%, ultrafine mineral powder accounted for 5%, naphthalene water reducing agent accounted for 3~5‰, and lignin sulfonate calcium accounted for 1~3‰. The effects of water–cement ratio and water reducer content on slurry viscosity and water bleeding rate were tested by laboratory experiments. Based on the fracture characteristics of surrounding rock and the "Liu Jiacai Formula", the influence law of fracture opening, grouting pressure and slurry viscosity on the slurry diffusion radius was analyzed. The results show that the slurry viscosity decreases with the increase of water–cement ratio and water reducer content, but the bleeding rate increases obviously with the increase of the two factors; when the water–cement ratio is 1.0 and the water reducer content is 3‰, the slurry has the advantages of "strong permeability, strong flow and low water bleeding rate"; the smaller the fracture opening is, the greater the required grouting pressure and the lower the required slurry viscosity. Aiming at the "micro-fracture zone" of surrounding rock in deep roadways, when the dynamic viscosity of the slurry is 2.0 mPa·s, the reasonable grouting pressure should be 12 MPa to meet the needs of grouting reinforcement engineering. The high-pressure grouting test of surrounding rock in the "micro-fracture zone" was successfully carried out by using the superfine cement-based composite grouting material.

Keywords: deep roadway; fractured surrounding rock; superfine cement; slurry fluidity; slurry diffusion radius

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1. Introduction

As the main energy source in China, coal reserves with a depth of more than 600 m account for 73% [1]. With the decrease of shallow coal resources, China's coal mines gradually turn to deep mining, and the mining depth has reached 1000–1500 m [2]. Under the high stress in the deep strata, the fractures of soft surrounding rock of roadways are compressed and closed, presenting a state of "micro-fractures and low permeability" [3,4], making it difficult to be grouted [5–9].

The performance of grouting materials is an important factor affecting the reinforcement effect of surrounding rock [10–13]. Grouting materials mainly include chemical materials and cement-based materials. Although the chemical material has good permeability, it has the defects of low strength, high price, and environmental pollution. At present, cement-based grouting materials are commonly used in coal mine roadways. Ordinary cement has a large particle size (30–45 μm) and poor groutability, and can only inject fractures with a fracture opening greater than 0.1 mm, while superfine cement with a particle size smaller than 10 μm can inject micro-fractures with fracture openings of less than 0.1 mm [10]. Superfine cement slurry has the advantages of good permeability, high consolidation strength, low price, and no pollution, which provides an effective solution for the grouting of micro-fracture surrounding rock in deep roadways [14,15].

In recent years, many scholars have studied the performance and diffusion law of superfine cement slurry, and applied it to the grouting project of roadways, which improved the reinforcement effect of surrounding rock [14–23]. Guan et al. and Wang et al. studied the particle size distribution, fluidity, setting time and consolidation strength of superfine cement-based grouting materials [14,15]. Yu et al. and Guo et al. studied the fluidity and stability of superfine cement slurry, and applied it to the grouting engineering of roadway-surrounding rock [16,17]. Wu et al. and He et al. studied the diffusion law of superfine cement slurry and carried out field grouting reinforcement test [18,19].

At present, although the application of superfine cement is gradually increasing, there are still the following problems for grouting reinforcement in deep roadways: ① Although the superfine cement has a small particle size and good permeability, the superfine cement slurry has the defects of "easy agglomeration, high viscosity and more water precipitation". ② The groutability of the micro-fractures of surrounding rock is poor, the diffusion law of slurry in micro-fractured surrounding rock is still unclear, and the selection of grouting parameters is still blind. ③ The grouting pressure used in coal mine roadways is generally below 6~8 MPa, and the grouting pressure is low, so it is difficult to inject into the micro-fractured surrounding rock. Based on the above problems of grouting reinforcement of micro-fractured surrounding rock in deep roadways, we developed a superfine cement-based composite grouting material, and studied the slurry's flow characteristics and its diffusion law. The research results can provide an important reference for the selection of reasonable grouting parameters in deep roadways.

2. Composition Optimization of Superfine Cement-Based Composite Grouting Material

Superfine cement generally refers to cement with a particle size grading of D90 < 20 μm, and its fineness is generally above 600 mesh [14]. Due to the small particle size of superfine cement, its permeability has been greatly improved, but there are some defects of "easy agglomeration, high viscosity, and more water precipitation" when configuring the slurry. For example, in order to ensure the fluidity of the slurry, when the slurry is configured with a water–cement ratio of 1.5~2:1, the water bleeding rate reaches 30~40%. In order to overcome the above defects, a superfine cement-based composite grouting material was developed by adding preferred excipients and additives with ultra-fine cement as the main component. The composition and characteristics of the composite grouting material are shown in Figure 1.

(1) Superfine cement. The superfine cement of 800 mesh or more with the particle size of D90 < 15 and D50 < 5.5 μm was selected, which has the characteristics of small particle size and large specific surface area. By reducing the particle size, the groutability of the cement slurry in fractured surrounding rock can be enhanced. According to the principle of the groutability of the fractured rock, "the fracture width should be greater than three times the particle size of the grouting material", the width of the injectable fracture should be less than 16.5 μm.

(2) Superfine fly ash. It is obtained by grinding low-calcium fly ash. The particles are spherical microbeads with a smooth surface and a regular shape. The particle size is 1~5 μm. The particles are well-graded and have good morphology and activity. Ultra-fine fly ash has a significant effect on the dispersion of cement particles in the slurry [20]. Adding

superfine fly ash to superfine cement slurry can play a role in "improving workability, reducing agglomeration and improving fluidity".

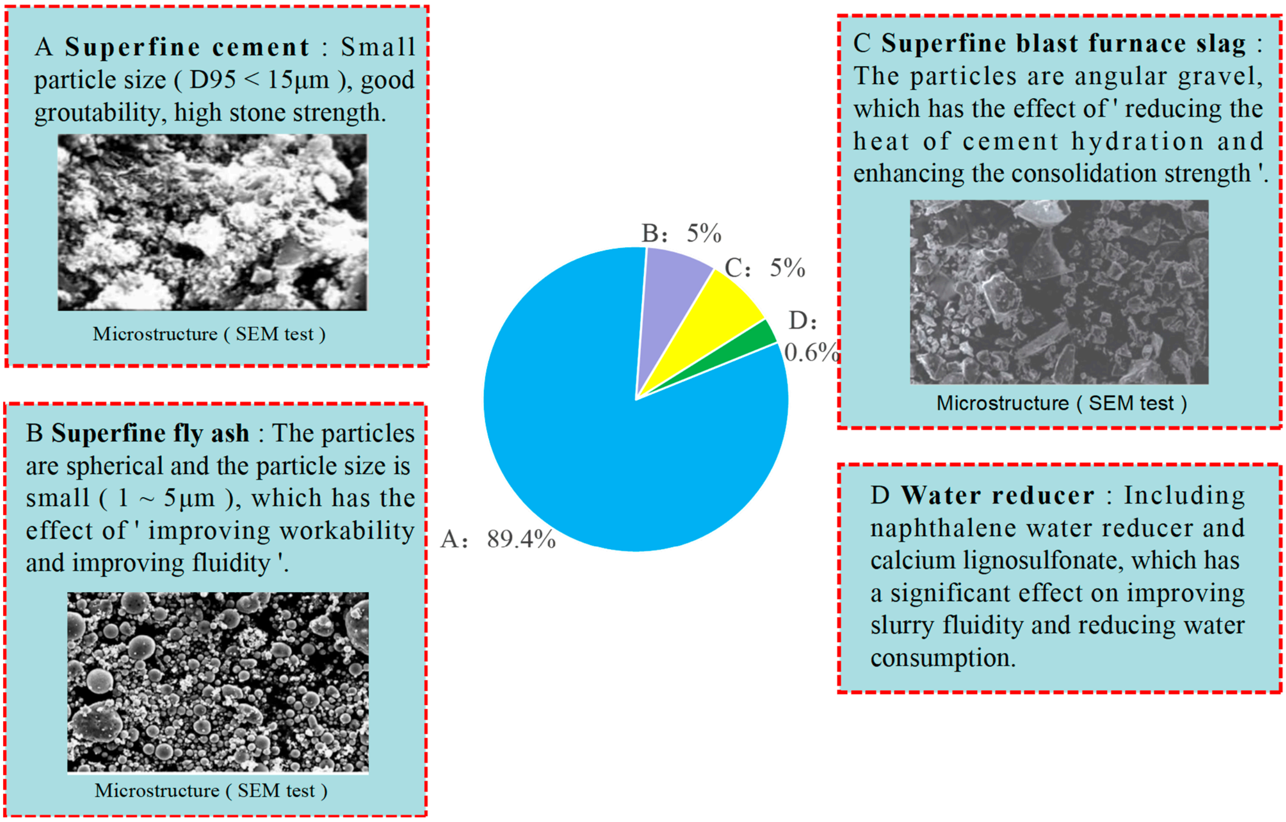


Figure 1. Composition and characteristics of superfine cement-based composite grouting material: A: superfine cement; B: superfine fly ash; C: superfine blast furnace slag; D: water reducer.

(3) Superfine blast furnace slag. The particles of blast furnace slag are micro-gravel-like with different shapes and sharp edges [20], and the particle size is D90 < 15 μm. Adding a certain proportion of superfine blast furnace slag into the superfine cement can improve the cementation and consolidation compactness of the slurry, and has the effect of "reducing the hydration heat of cement and enhancing the strength of the consolidated body".

(4) Composite water reducer, including naphthalene water reducer and calcium lignosulfonate. Naphthalene water reducer is an efficient water reducer, which can effectively improve the fluidity of the slurry and reduce water consumption, and is often used to configure low-viscosity and strong-penetration slurry. Calcium lignosulfonate is cheap and can be used together with naphthalene water reducer.

Through the analysis of composition characteristics, the silicate cement with a particle fineness of 800 mesh and a strength grade of 52.5 was selected as the basic material, which was mixed with superfine fly ash, superfine blast furnace slag, naphthalene water reducer, and calcium lignosulfonate to prepare superfine cement-based composite grouting material. The optimized grouting material components are as follows: superfine cement accounts for 89.4%, superfine fly ash accounts for 5%, superfine mineral powder accounts for 5%, naphthalene water reducing agent accounts for 3~5‰, and calcium lignosulfonate accounts for 1~3‰; the particle size of the composite grouting material is D90 < 15 μm.

3. Slurry Flow Characteristics and Ratio Optimization of Superfine Cement-Based Composite Grouting Material

3.1. Experimental Scheme Design

In this experiment, superfine cement-based composite grouting material of 800 mesh was used as the experimental material, and the slurry flow characteristics were tested under different water–cement ratios and water reducer contents. In the experiment, the water–cement ratio was selected as 0.8, 1.0, and 1.2, and the amount of naphthalene water reducer was selected as 3‰, 5‰, 7‰, and 9‰ of the superfine cement weight. By comparing the slurry viscosity and water bleeding rate under different combinations, the reasonable water–cement ratio and water reducer content were determined. Experimental materials and test instruments are shown in Figure 2. The test scheme design is shown in Table 1.

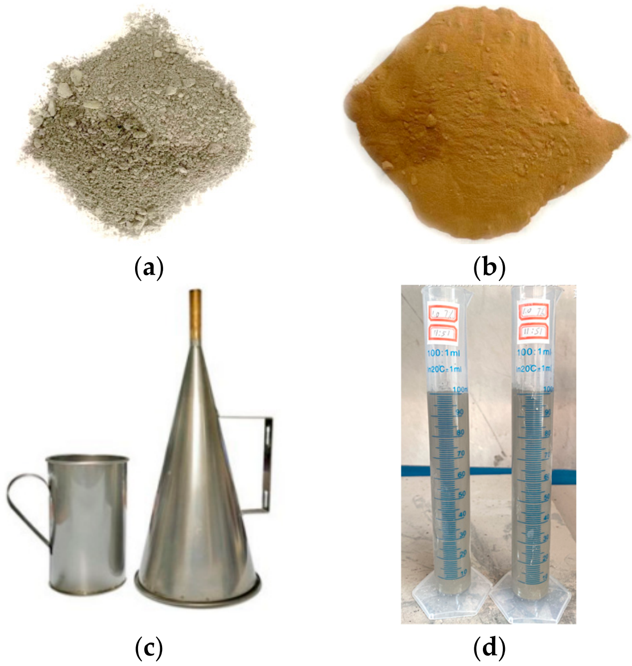


Figure 2. Experimental materials and test instruments: (**a**) superfine cement; (**b**) naphthalene water reducer; (**c**) Marsh funnel viscosimeter; (**d**) test cylinder of water bleeding rate.

Table 1. Test scheme of slurry flow characteristic of superfine composite grouting material.

Water–Cement Ratio	Water Reducer Content			
0.8	3‰	5‰	7‰	9‰
1.0	3‰	5‰	7‰	9‰
1.2	3‰	5‰	7‰	9‰

Viscosity measurement: the viscosity of the slurry was measured using an MLN-2 Marsh funnel viscometer. The test of Marsh viscosity was carried out according to the "Petroleum and natural gas industries—field testing of drilling fluids"(GB/T 16783.1-2014). The measured viscosity standard value is that the time during which 946 mL standard distilled water flows out at 20 ± 2 °C is 26 ± 0.5 s.

Determination of water bleeding rate: after preparing the grout according to different water–cement ratios and water reducer contents, the slurry was poured into a 100 mL measuring cylinder. After the slurry condensation tended to be stable, the water bleeding rate of the slurry was observed.

3.2. Analysis of Slurry Viscosity

Slurry viscosity is a key index to measure slurry fluidity. Based on the experimental results in Table 2, the relationship between Marsh viscosity and water–cement ratio and water reducer content was obtained, as shown in Figure 3.

Table 2. Slurry viscosity of different water–cement ratios and water reducer contents.

Water–Cement Ratio	Marsh Viscosity/s			
	Water Reducer Content 3‰	Water Reducer Content 5‰	Water Reducer Content 7‰	Water Reducer Content 9‰
0.8	44.71	39.88	38.23	36.98
	44.21	39.71	38.45	36.48
Average	44.46	39.79	38.34	36.73
1.0	33.99	32.62	31.71	31.04
	34.40	32.83	31.57	30.89
Average	34.19	32.72	31.64	30.69
1.2	30.65	29.92	28.75	28.65
	30.35	29.76	29.18	28.31
Average	30.50	29.84	28.96	28.48

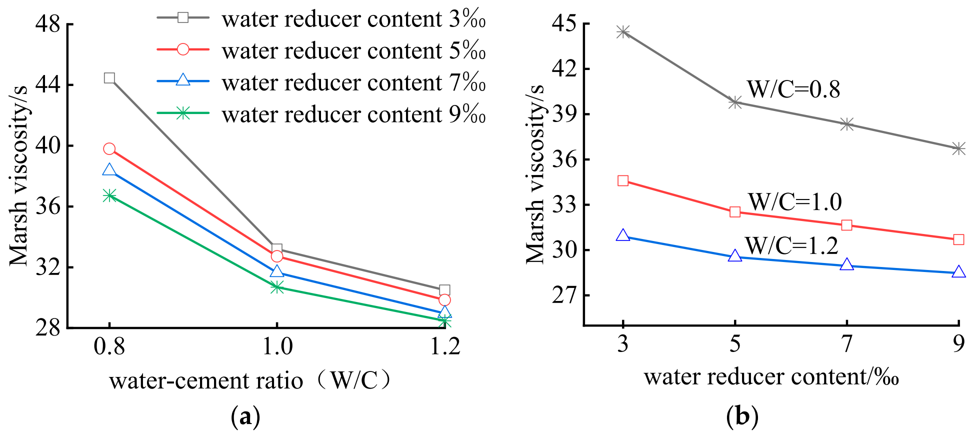


Figure 3. Relationship between Marsh viscosity and water–cement ratio and water reducer content: (**a**) Marsh viscosity and water–cement ratio; (**b**) Marsh viscosity and water reducer content.

(1) The water–cement ratio had a great influence on the slurry viscosity: when the water reducer content was constant, the Marsh viscosity decreased significantly with the increase of the water–cement ratio (Figure 3a), but when the water–cement ratio was greater than 1.0, its decline gradually slowed down. For example, when the water reducer content was 3‰ and the water–cement ratio was 0.8, 1.0, and 1.2, the slurry viscosity was 44.46, 34.19, and 30.50 s, respectively. It can be seen that when the water–cement ratio was greater than 1.0, the Marsh viscosity only decreased by 3.69 s.

(2) The water reducer content had relatively little influence on the viscosity of the slurry: when the water–cement ratio was constant, the Marsh viscosity decreased with the increase of water reducer content (Figure 3b). The larger the water–cement ratio was, the smaller was the influence of water reducer content on Marsh viscosity. For example, when the water–cement ratio reached 1.2, the Marsh viscosity only decreased by 2.02 s when the water reducer content increased from 3‰ to 9‰.

(3) When the water–cement ratio was 1.0 and the water reducer content was 3‰~9‰, the grout was easy to stir and no agglomerating phenomenon occurred. The Marsh viscosity was 34.19~30.69 s, the slurry viscosity was small, and the fluidity was good.

3.3. Analysis of Water Bleeding Rate

Water bleeding rate is an important index reflecting the stability of a slurry. Based on the experimental results in Table 3, the relationship between water bleeding rate and water–cement ratio and water reducer content was obtained, as shown in Figure 4.

Table 3. Water bleeding rate of different water–cement ratios and water reducer contents.

Water–Cement Ratio	Water Bleeding Rate/%			
	Water Reducer Content 3‰	**Water Reducer Content 5‰**	**Water Reducer Content 7‰**	**Water Reducer Content 9‰**
0.8	2.0	2.0	7.0	11.0
	1.0	3.0	6.0	10.0
average/%	1.5	2.5	6.5	10.5
1.0	5.0	9.0	13.0	17.0
	5.0	8.0	12.0	16.0
average/%	5.0	8.5	12.5	16.5
1.2	19.0	20.0	22.0	22.0
	20.0	21.0	21.0	22.0
average/%	19.5	20.5	21.5	22.0

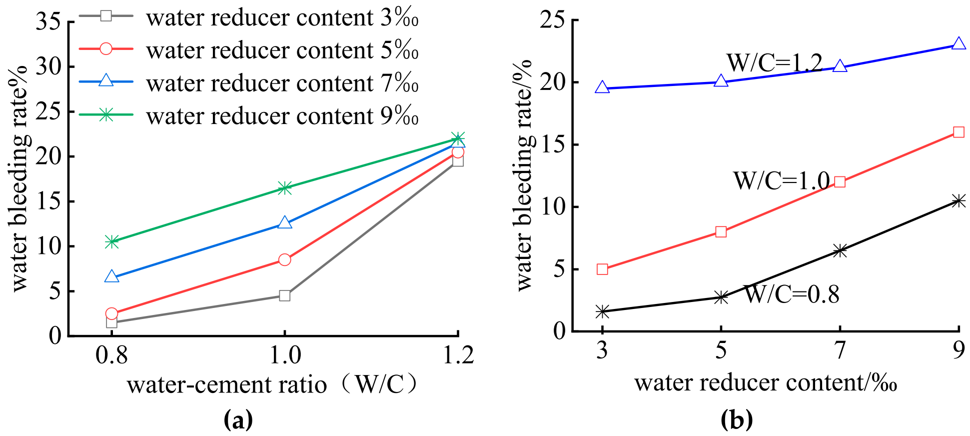


Figure 4. Relationship between water bleeding rate and water–cement ratio and water reducer content: (**a**) water bleeding rate and water–cement ratio; (**b**) water bleeding rate and water reducer content.

(1) When the water reducer content was constant, the water bleeding rate increased significantly with the increase of water–cement ratio (Figure 4a). For example, when the water reducer content was 3‰ and the water–cement ratio was 0.8, 1.0, and 1.2, the water bleeding rate of the slurry was 1.5%, 5.0%, and 19.5%, respectively. When the water–cement ratio was greater than 1.0, the water bleeding rate increased significantly; when the water–cement ratio was 1.2, the water bleeding rate increased to 19.5%. An excessive water bleeding rate will cause the surrounding rock to be weakened, which is not conducive to the stability of roadway-surrounding rock.

(2) When the water–cement ratio was constant, the water bleeding rate of grout increased with the increase of water reducer content (Figure 4b), and the smaller the water–cement ratio was, the greater was the influence of the water reducer content on the water bleeding rate of grout. When the water–cement ratio was 1.0 and the water reducer content was 3‰, 5‰, 7‰, and 9‰, the water bleeding rate was 5.0%, 8.5%, 12.5%, and 16.5%, respectively. When the water reducer content is greater than a certain value between 5‰ and 7‰, the water bleeding rate will exceed 10%, which is not conducive to the stability of roadway-surrounding rock.

Considering the two factors of slurry viscosity and water bleeding rate, the reasonable water–cement ratio of a superfine cement-based composite slurry is 1.0 and the water reducer content is 3‰. Under these conditions, the Marsh viscosity is 34.19 s and the water bleeding rate is 5%. The slurry has the advantages of “good permeability, no agglomeration, low viscosity, and low water bleeding rate”, so its groutability is better.

4. Slurry Diffusion Law of Superfine Cement-Based Composite Grouting Material

Based on the superfine cement-based composite grouting material, the influence law of fracture opening, grouting pressure, and slurry viscosity on the diffusion radius of the slurry was studied.

4.1. Influencing Factors of Slurry Diffusion Radius

The slurry diffusion radius is an important index to measure the groutability of grouting materials. For grouting in fractured rock masses, Liu Jiacai proposed the radial flow diffusion equation of slurry under parallel fracture conditions, which is the famous "Liu Jiacai Formula" [18]:

$$R = \sqrt[2.21]{\frac{0.093(P - P_w)Tb^2 r_0^{0.21}}{\mu}} + r_0 \tag{1}$$

where R is the slurry diffusion radius, cm; r_0 is the radius of grouting hole, cm; μ is the slurry dynamic viscosity, Pa·s; P is the grouting pressure, MPa; P_W is the pressure of groundwater in the fracture, MPa; b is the fracture opening, cm; and T is the grouting time, s.

According to the "Liu Jiacai Formula", the main influencing factors of grout diffusion radius include fracture opening, grouting pressure and slurry viscosity. Among them, fracture opening is the objective basic condition, while grouting pressure and slurry viscosity are the key factors that can be controlled artificially.

(1) Fracture opening. Fracture is the channel of slurry diffusion, and its opening has a great influence on the slurry diffusion radius. After the excavation of deep roadways, the surrounding rock forms a "fracture development zone, micro-fracture zone and fracture closure zone" from shallow to deep, as shown in Figures 5 and 6. In the fracture development zone, the fracture opening is generally greater than 0.1 mm, and some fractures opening can be more than 1 mm, and its groutability is good. In the micro-fracture zone, the fracture opening is small, generally less than 0.1 mm, and the ordinary cement slurry is difficult to inject, so a superfine cement-based composite grouting material with good groutability is needed. In the fracture closure zone, the fractures is in a closed state, which is a non-injection zone.

(2) Grouting pressure. Grouting pressure is the driving force of slurry diffusion. The slurry diffusion radius is positively correlated with the grouting pressure. For the surrounding rock of the "fracture development zone", the fracture opening is large, and the grouting pressure required is generally less than 5 MPa. For the "micro-fracture zone", the fracture opening is small and the grout diffusion resistance is large. Under the conventional grouting pressure of 6–8 MPa, the diffusion radius is small, which makes it difficult to meet the needs of grouting.

(3) Slurry viscosity. Slurry viscosity is an important index to measure slurry fluidity, and it has a great influence on the diffusion radius of the slurry. When calculating the diffusion radius of the slurry, the dynamic viscosity is generally used instead of the Marsh viscosity. M.J. Pitt. and Liu H.j. studied the relationship between the dynamic viscosity and the Marsh viscosity [21]:

$$t = t_v \left[1 + x_1 \left(\frac{\mu}{\rho} \cdot \frac{t_v}{A} \right)^{1.2} \right] \tag{2}$$

where t is the Marsh viscosity, and the superfine cement-based composite grouting material is adopted. When the water–cement ratio is 0.8, 1.0, and 1.2, and the water reducer content is 3‰, the Marsh viscosity is 44.46, 34.19, and 30.50 s, respectively; t_v is the outflow time of distilled water under standard conditions, 26 s; A is the cross-sectional area of the MLN-2 funnel nozzle, 1.54 cm^2; ρ is the grout density, and when the water–cement ratio is 0.8, 1.0, and 1.2, grout density is 1.8, 1.7, and 1.45 g/cm^3, respectively; x_1 is the digital eigenvalue of the standard funnel, 0.016; and μ is the dynamic viscosity of slurry, mPa·s. The bulk density of the tested sample of superfine cement-based composite grouting material is 3.1 g/cm^3.

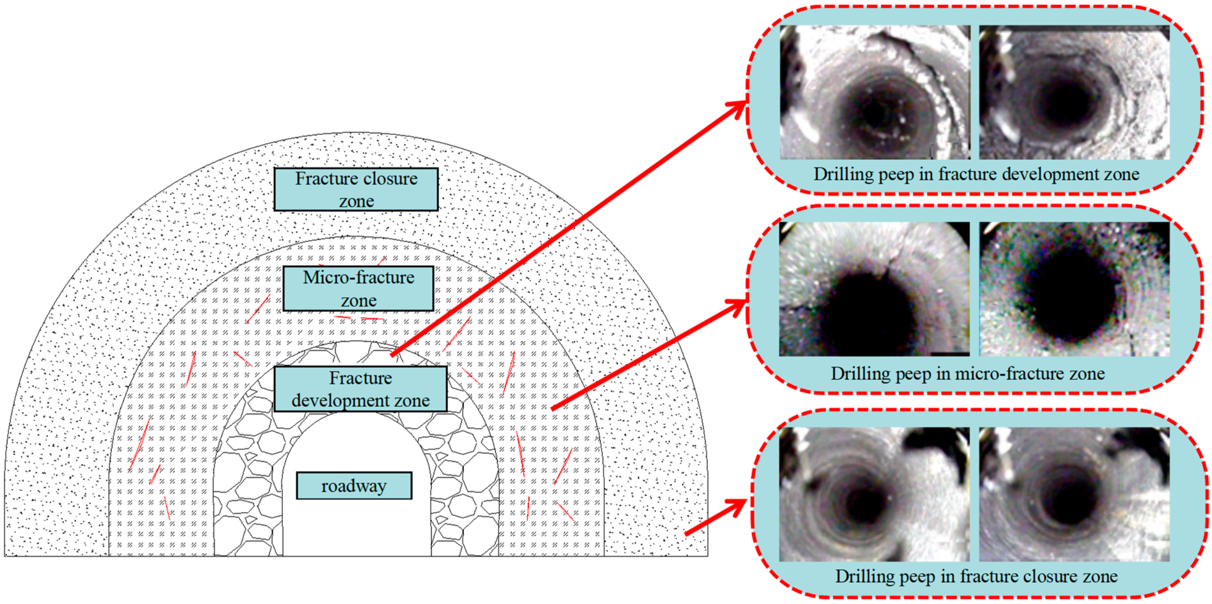


Figure 5. Fracture partition diagram of surrounding rock in deep roadways.

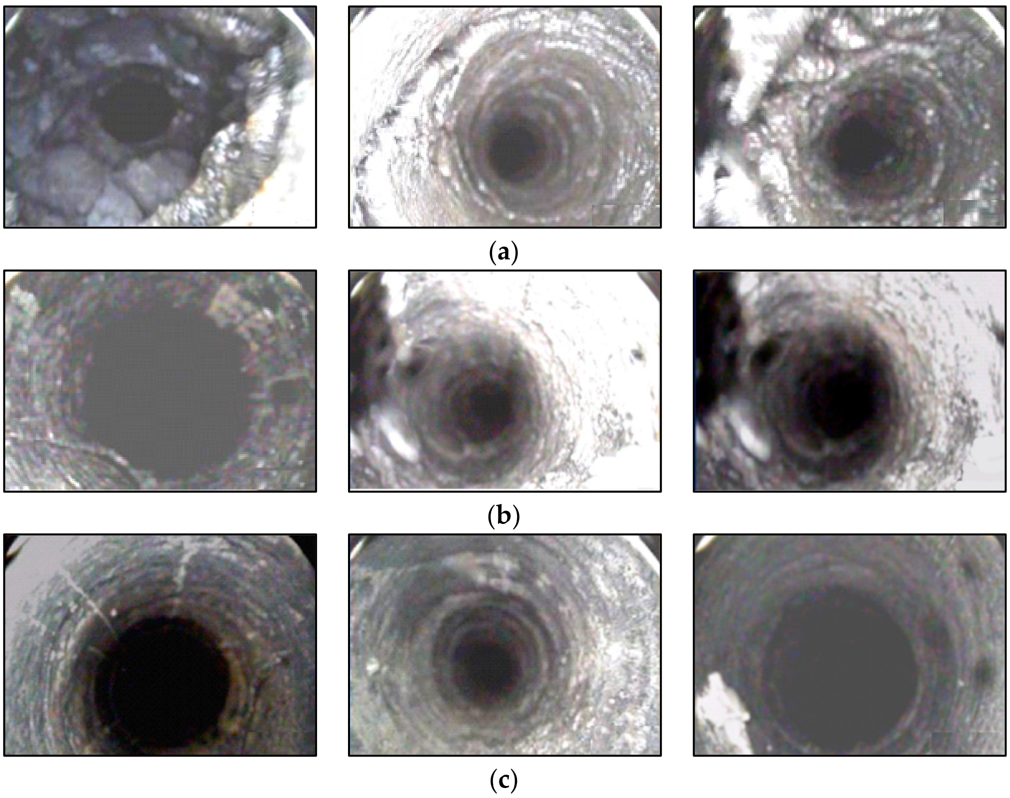


Figure 6. Typical borehole peep maps: (**a**) fracture development zone; (**b**) micro-fracture zone; (**c**) fracture closure zone.

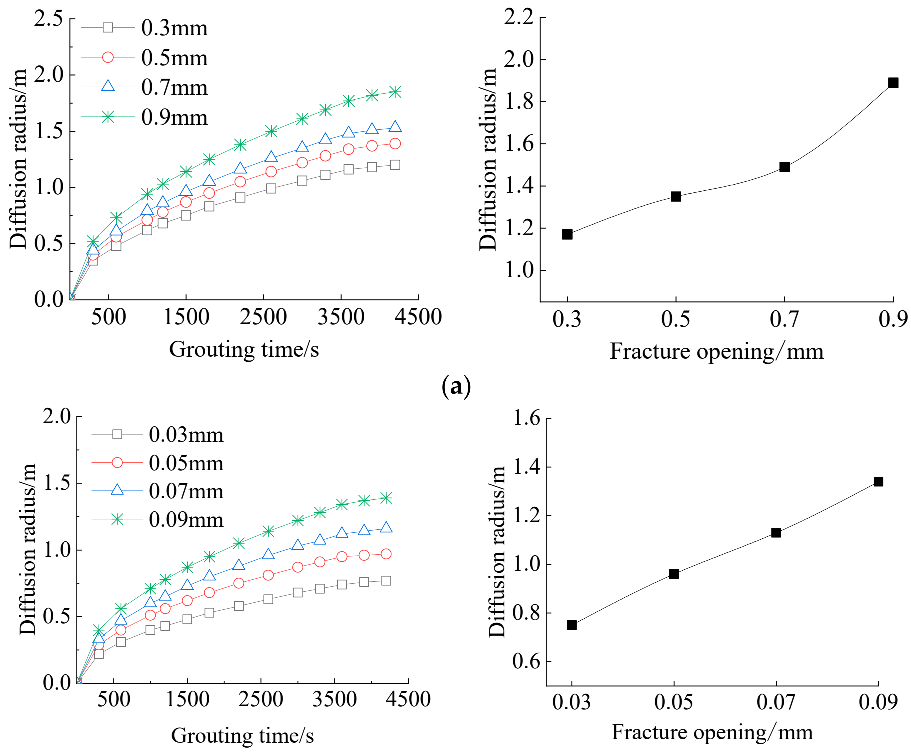
By substituting the above parameters into Equation (2), it can be obtained through calculation that when the water–cement ratio is 0.8, 1.0, and 1.2, and the water reducer content is 3‰, the slurry dynamic viscosity is 4.6, 2.0, and 1.1 mPa·s, respectively.

4.2. Relationship between Slurry Diffusion Radius and Fracture Opening

For the superfine cement-based composite grouting material, the "Liu Jiacai Formula" was used to calculate the slurry diffusion radius under different fracture openings. The values of variables in Formula (1) were as follows: r_0 is 15 mm, P_W is 0 Mpa, and μ is 2.0 mPa·s; for the "fracture development zone" of surrounding rock, P was set as 3 MPa, and b was set as 0.3, 0.5, 0.7, and 0.9 mm; for the "micro-fracture zone" of surrounding rock, P was set as 12 MPa, and b was set as 0.03, 0.05, 0.07, and 0.09 mm. Under the above conditions, the slurry diffusion radius R under different fracture openings was calculated, as shown in Table 1. The relationship between the slurry diffusion radius R and fracture openings b was obtained by analyzing the data in Table 1, as shown in Figure 7. It can be seen from Table 4 and Figure 7 that:

(1) When P is fixed, the slurry diffusion radius R increases with the increase of fracture openings b. When b is 0.3, 0.5, 0.7, and 0.9 mm and T is 3600 s, R is 1.16, 1.34, 1.48, and 1.89 m, respectively. When b is 0.03, 0.05, 0.07, and 0.09 mm and T is 3600 s, R is 0.74, 0.95, 1.12, and 1.34 m, respectively.

(2) Compared with the "fracture development zone", the fracture opening of the "micro-fracture zone" is smaller, and the grouting pressure required to reach the same diffusion radius is also greatly improved: when b is 0.3 mm, and the grouting pressure is 3 Mpa and the slurry diffusion radius can reach more than 1 m, while when b is 0.07 mm, the grouting pressure should be increased to 12 MPa.



(**a**) (**b**)

Figure 7. Relationship between slurry diffusion radius and fracture opening: (**a**) grouting pressure in development fracture zone is 3 MPa; (**b**) grouting pressure in micro-fracture zone is 12 MPa.

Table 4. Slurry diffusion radius under different fracture openings.

Serial Number	Grouting Time *T* (s)	When *P* Is 3 MPa, Slurry Diffusion Radius under Different Fracture Openings *R* (m)				Grouting Time *T* (s)	When *P* Is 12 MPa, Slurry Diffusion Radius under Different Fracture Openings *R* (m)			
		0.3 mm	0.5 mm	0.7 mm	0.9 mm		0.03 mm	0.05 mm	0.07 mm	0.09 mm
1	300	0.35	0.40	0.44	0.56	300	0.22	0.29	0.33	0.40
2	600	0.48	0.56	0.61	0.78	600	0.31	0.40	0.47	0.56
3	1000	0.62	0.71	0.79	1.00	1000	0.40	0.51	0.60	0.71
4	1200	0.68	0.78	0.86	1.10	1200	0.43	0.56	0.65	0.78
5	1500	0.75	0.87	0.96	1.22	1500	0.48	0.62	0.73	0.87
6	1800	0.83	0.95	1.05	1.34	1800	0.53	0.68	0.80	0.95
7	2200	0.91	1.05	1.16	1.48	2200	0.58	0.75	0.88	1.05
8	2600	0.99	1.14	1.26	1.60	2600	0.63	0.81	0.96	1.14
9	3000	1.06	1.22	1.35	1.72	3000	0.68	0.87	1.03	1.22
10	3300	1.11	1.28	1.42	1.81	3300	0.71	0.91	1.07	1.28
11	3600	1.16	1.34	1.48	1.89	3600	0.74	0.95	1.12	1.34
12	3900	1.18	1.37	1.51	1.91	3900	0.76	0.96	1.14	1.37
13	4200	1.20	1.39	1.53	1.91	4200	0.77	0.97	1.16	1.39

4.3. Relationship between Slurry Diffusion Radius and Grouting Pressure

In Formula (1), for the "fracture development zone", *b* was set as 0.3 mm, and *P* was set as 1, 3, 5, and 7 MPa; for the "micro-fracture zone", *b* was set as 0.07 mm, and *P* was set to 6, 8, 10, 12 MPa; the remaining parameters are the same as above. According to the "Liu Jiacai Formula", the slurry diffusion radius under different grouting pressures was calculated, as shown in Table 2. The relationship between the slurry diffusion radius and the grouting pressure was obtained by analyzing the data in Table 2, as shown in Figure 7. It can be seen from Table 5 and Figure 8 that when the fracture opening is constant, the slurry diffusion radius increases with the increase of grouting pressure:

(1) For the "fracture development zone", when *P* is 1, 3, 5, 7 MPa and *T* is 3600 s, the slurry diffusion radius is 0.68, 1.16, 1.49, 1.77 m respectively. It can be seen that when the grouting pressure is ≥3 MPa, the slurry diffusion radius is >1 m. Considering that excessive grouting pressure will cause the shallow surrounding rock of the roadways to bulge or destroy, the reasonable grouting pressure is designed to be 3 MPa.

(2) For the "micro-fracture zone", when *P* is 6, 8, 10, 12 MPa and *T* is 3600 s, the slurry diffusion radius is 0.80, 0.92, 1.03, 1.12 m respectively. It can be seen that only when the grouting pressure is above 10 MPa, the slurry diffusion radius is more than 1 m. Considering the heterogeneity of the surrounding rock and the pressure loss of the grouting pipeline, the surplus coefficient is selected as 1.2, and the actual grouting pressure is designed to be 12 MPa for the engineering practice.

Table 5. Slurry diffusion radius under different grouting pressures.

Serial Number	Grouting Time *T* (s)	When *b* Is 0.3 mm, Slurry Diffusion Radius under Different Grouting Pressure (m)				Grouting Time *T* (s)	When *b* Is 0.07 mm, Slurry Diffusion Radius under Different Grouting Pressure (m)			
		1 MPa	3 MPa	5 MPa	7 MPa		6 MPa	8 MPa	10 MPa	12 MPa
1	300	0.21	0.35	0.45	0.52	300	0.24	0.28	0.31	0.33
2	600	0.29	0.48	0.63	0.73	600	0.33	0.38	0.43	0.47
3	1000	0.36	0.62	0.79	0.94	1000	0.43	0.49	0.55	0.60
4	1200	0.40	0.68	0.87	1.03	1200	0.47	0.54	0.60	0.65
5	1500	0.44	0.75	0.97	1.14	1500	0.52	0.60	0.67	0.73
6	1800	0.48	0.83	1.06	1.25	1800	0.57	0.65	0.73	0.80
7	2200	0.53	0.91	1.17	1.38	2200	0.63	0.72	0.80	0.88
8	2600	0.56	0.95	1.22	1.44	2600	0.65	0.75	0.84	0.96
9	3000	0.62	1.06	1.37	1.61	3000	0.73	0.84	0.94	1.03
10	3300	0.65	1.11	1.43	1.69	3300	0.76	0.88	0.98	1.07
11	3600	0.68	1.16	1.49	1.77	3600	0.80	0.92	1.03	1.12
12	3900	0.69	1.18	1.53	1.83	3900	0.84	0.94	1.05	1.15
13	4200	0.70	1.20	1.55	1.86	4200	0.86	0.96	1.07	1.18

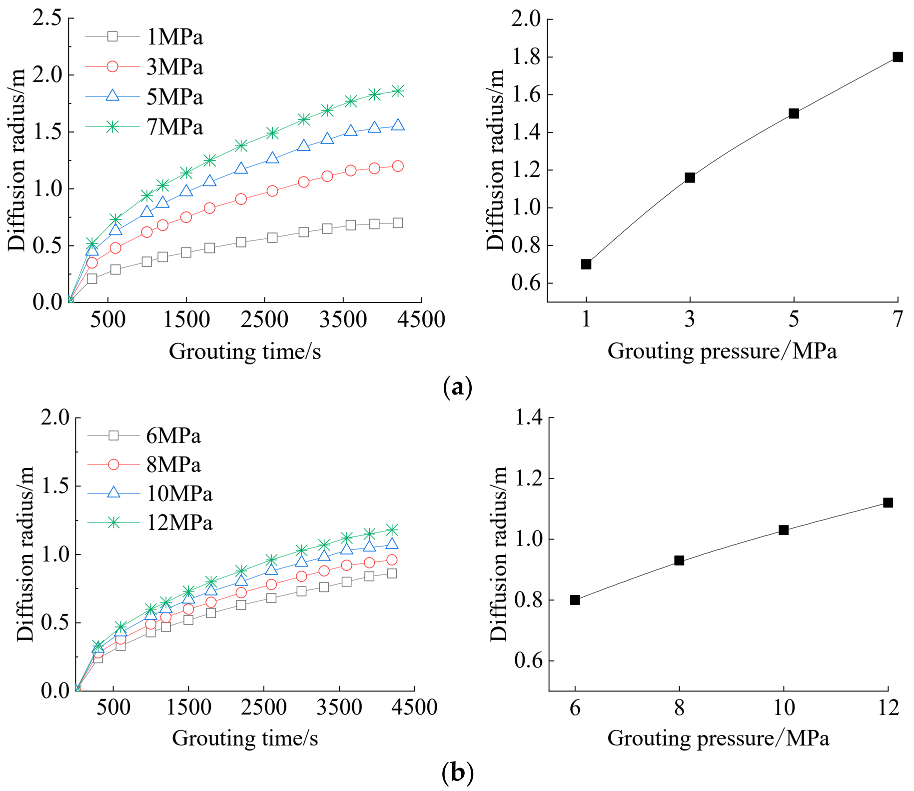


Figure 8. Relationship between slurry diffusion radius and grouting pressures: (**a**) Fracture opening 0.3 mm; (**b**) Fracture opening 0.07 mm.

4.4. Relationship between Slurry Diffusion Radius and Slurry Viscosity

In Formula (1), for the "fracture development zone", b was set as 0.3 mm, and P was set as 3 MPa; for the "micro-fracture zone", b was set as 0.07 mm, and P was set to 12 MPa; when the water–cement ratios were 0.8, 1.0, and 1.2, the values of μ were 4.6, 2.0, and 1.1 mPa·s, respectively. According to the " Liu Jiacai formula", the slurry diffusion radius under different slurry viscosities was calculated, as shown in Table 6.

Table 6. Slurry diffusion radius under different slurry viscosities.

Serial Number	Grouting Time (s)	When P Is 3 MPa, b Is 0.3 mm, Slurry Diffusion Radius under Different Slurry Viscosities R (m)			Grouting Time (s)	When P Is 12 MPa, b Is 0.07 mm, Slurry Diffusion Radius under Different Slurry Viscosities R (m)		
		4.6 mPa·s	2.0 mPa·s	1.1 mPa·s		4.6 mPa·s	2.0 mPa·s	1.1 mPa·s
1	300	0.20	0.35	0.42	300	0.16	0.33	0.41
2	600	0.27	0.48	0.59	600	0.22	0.47	0.57
3	1000	0.35	0.62	0.75	1000	0.29	0.60	0.73
4	1200	0.38	0.68	0.83	1200	0.31	0.65	0.80
5	1500	0.42	0.75	0.92	1500	0.35	0.73	0.89
6	1800	0.46	0.83	1.01	1800	0.38	0.80	0.97
7	2200	0.51	0.91	1.11	2200	0.42	0.88	1.07
8	2600	0.55	0.99	1.21	2600	0.45	0.96	1.17
9	3000	0.59	1.06	1.30	3000	0.48	1.03	1.25
10	3300	0.62	1.11	1.36	3300	0.51	1.07	1.31
11	3600	0.64	1.16	1.42	3600	0.53	1.12	1.37
12	3900	0.65	1.18	1.44	3900	0.55	1.15	1.39
13	4200	0.66	1.19	1.46	4200	0.56	1.16	1.41

The relationship between the slurry diffusion radius and the slurry viscosity was obtained by analyzing the data in Table 3, as shown in Figure 9. It can be seen from Table 3 and Figure 9 that when the fixed fracture opening and grouting pressure are constant, the slurry diffusion radius decreases with the increase of slurry viscosity:

(1) For the "fracture development zone", when μ is 4.6, 2.0, and 1.1 mPa·s and T is 3600 s, the slurry diffusion radius is 0.64, 1.16, and 1.42 m, respectively. When $\mu \leq 2.0$ mPa·s (water–cement ratio ≥ 1), the slurry diffusion radius is >1 m, and it can meet the needs of grouting engineering.

(2) For the "micro-fracture zone", when μ is 4.6, 2.0, and 1.1 mPa·s and T is 3600 s, the slurry diffusion radius is 0.53, 1.12, and 1.37 m, respectively. When $\mu \leq 2.0$ mPa·s (water–cement ratio ≥ 1), the slurry diffusion radius is >1 m. Considering that when the water–cement ratio is 1.2, the water bleeding rate is higher (19.5 %), which will cause the weakening of surrounding rock, the water–cement ratio was selected as 1.0.

In summary, the slurry diffusion radius is closely related to the fracture opening of the surrounding rock, grouting pressure, and slurry viscosity. The smaller the fracture opening, the greater the grouting pressure required. When the slurry dynamic viscosity of the superfine cement-based composite grouting material is 2.0 mPa·s (the water–cement ratio is 1.0), the reasonable grouting pressure should be 3 and 12 MPa respectively for the shallow "fracture development zone" (fracture opening > 0.1 mm) and deep "micro-fracture zone" (fracture opening $\leq$ 0.1 mm) in order to meet the grouting requirements of surrounding rock in deep roadways.

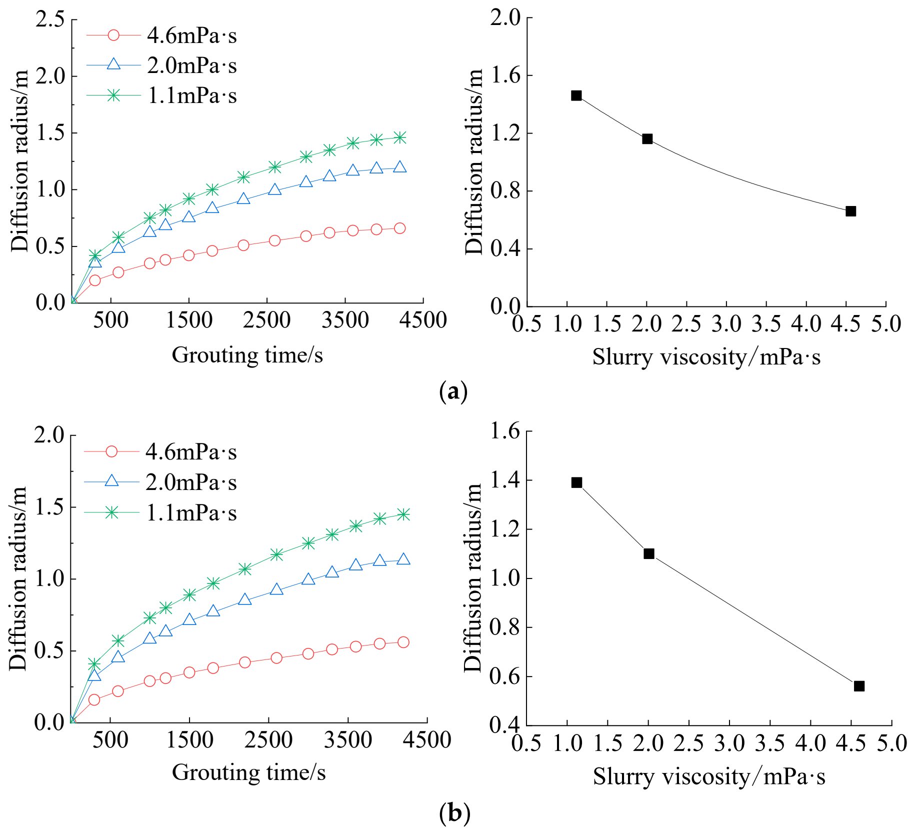


Figure 9. Relationship between slurry diffusion radius and slurry viscosity: (**a**) the grouting pressure is 3 MPa and the fracture opening is 0.3 mm; (**b**) the grouting pressure is 12 MPa and the fracture opening is 0.07 mm.

5. Engineering Application

Based on the slurry flow characteristics of superfine cement-based composite grouting material and its diffusion characteristics in the fractured surrounding rock, the application of grouting reinforcement in deep roadways was carried out.

5.1. Engineering Background

The buried depth of the 1103 roadway in Hudi Coal Mine is about 700 m, and the surrounding rock is mostly argillaceous rock with low strength. Under the action of high stress, the roadway showed serious deformation and failure. In the early stage, when the ordinary cement slurry and low grouting pressure were used, the "fracture development zone" had better injectability, but the "micro-fracture zone" was difficult to inject. Considering that the superfine cement-based composite grouting material has the advantages of "good permeability, no agglomeration, low viscosity, and low water bleeding rate", using this material for grouting reinforcement was carried out.

5.2. Grouting Scheme Design

Based on the slurry diffusion characteristics of the superfine cement-based composite grouting material, the grouting scheme of "3 MPa low-pressure grouting and 12 MPa high-pressure grouting" for the 1103 roadway was proposed, as shown in Figure 10.

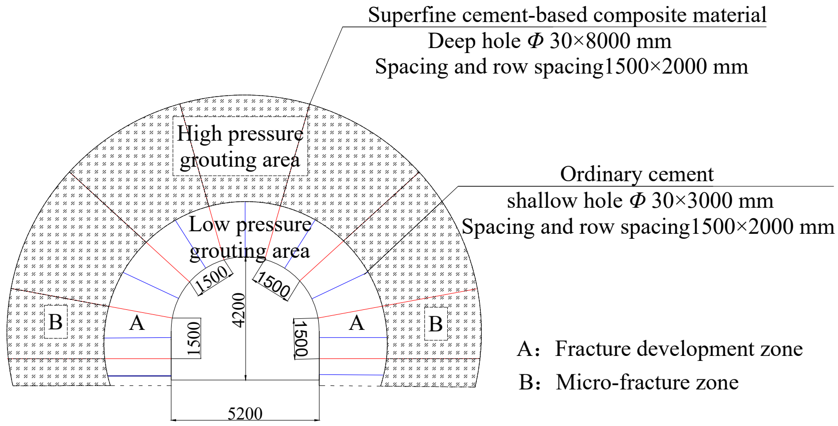


Figure 10. Grouting parameter layout of 1103 roadway.

(1) Three MPa low-pressure grouting for shallow surrounding rock: because the fracture opening of shallow surrounding rock was large, ordinary cement slurry was used for grouting. The grouting hole diameter was 30 mm, the hole depth was 3 m, the grouting pressure was 3 MPa, and the spacing and row spacing of grouting holes were 1500 and 2000 mm, respectively.

(2) Twelve MPa high-pressure grouting for deep surrounding rock: because the fracture opening of deep surrounding rock was small and the injectability was poor, the superfine cement-based composite grouting material was adopted. The water–cement ratio of the slurry was 1.0, and the water reducer content was 3‰. The grouting hole diameter was 30 mm, the hole depth was 8 m, the grouting pressure was 12 MPa, and the spacing and row spacing of grouting holes were 1500 and 2000 mm, respectively.

5.3. Grouting Reinforcement Effect

Based on the above technical parameters of grouting reinforcement, field tests were carried out in the 1103 roadway. In the grouting project, in order to achieve a grouting pressure of 12 MPa, a high-pressure grouting pump with a rated working pressure of 30 MPa and a hole sealer with a high-pressure hose expansion type were used. On the basis of low pressure grouting of shallow surrounding rock, high pressure grouting of deep surrounding rock could be realized.

After grouting in the 1103 roadway, the slurry diffusion effect was observed using a peephole, as shown in Figure 11. It can be seen that the fractures of surrounding rock were filled with gray-white slurry, indicating that the slurry diffusion effect is good and can play a good consolidation role. The field observation results show that the stability of surrounding rock of the deep roadway was significantly improved by using the superfine cement-based composite grouting material and the grouting scheme.

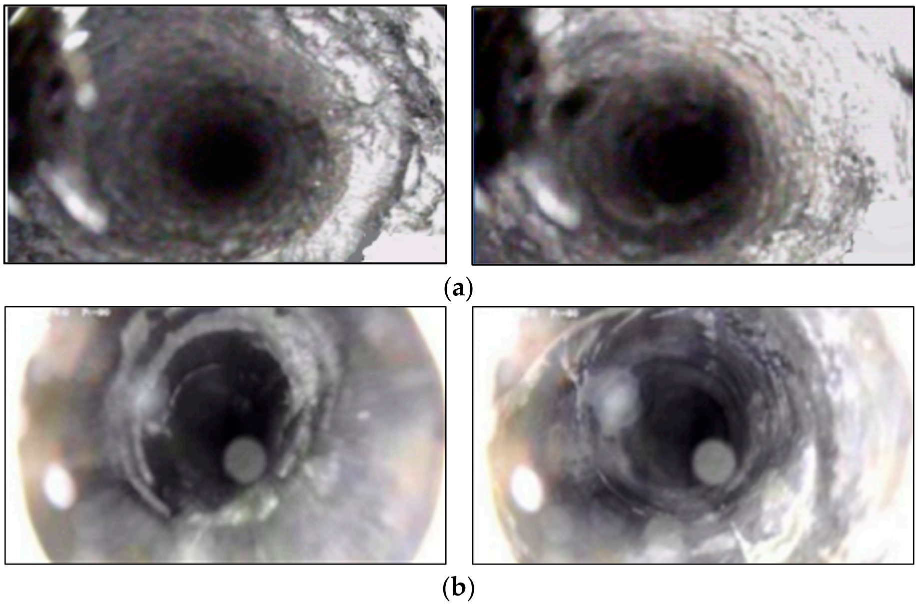


Figure 11. Slurry diffusion condition observed through peephole: (**a**) borehole peeping before grouting; (**b**) borehole peeping after grouting.

6. Conclusions

(1) The single superfine cement slurry has the disadvantages of "easy agglomeration, high viscosity and more water precipitation". By adding superfine fly ash, superfine blast furnace slag, naphthalene water reducer, and calcium lignosulfonate, the superfine cement-based composite grouting material is formed, which can effectively improve the workability and fluidity of the slurry and reduce the water bleeding rate of the slurry.

(2) The water–cement ratio has a great influence on the viscosity of the slurry, while the influence of water reducer content on it is relatively small, especially when the water–cement ratio is greater than 1. The water–cement ratio and the water reducer content have great influence on the water bleeding rate. Considering the two factors of slurry viscosity and water bleeding rate, the reasonable water–cement ratio of the composite grouting material is 1.0, and the reasonable water reducer content is 3‰. The slurry has the advantages of "good permeability, no agglomeration, low viscosity, and low water bleeding rate".

(3) The slurry diffusion radius is closely related to the fracture opening of the surrounding rock, grouting pressure and slurry viscosity. The smaller the fracture opening, the greater the grouting pressure required. When the slurry dynamic viscosity of the superfine cement-based composite grouting material is 2.0 mPa·s (the water–cement ratio is 1.0), the reasonable grouting pressures should be 3 MPa and 12 MPa respectively for the shallow "fracture development zone" and deep "micro-fracture zone" in order to meet the grouting requirements of surrounding rock in deep roadways.

(4) The grouting practice of surrounding rock in a deep roadway was carried out by using the superfine cement-based composite grouting material. Through the implementation of "3 MPa low-pressure grouting and 12 MPa high-pressure grouting", the slurry diffusion and consolidation effect were effectively improved.

(5) In this article, the flow characteristics of superfine cement-based composite materials and their diffusion law in fractured surrounding rock of deep roadways are studied, but the mechanical characteristics of consolidation body between slurry and surrounding rock are not studied. In the future, the mechanical characteristics of consolidation body will be studied by a rock mechanics experiment system.

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References

1. Bai, J.B.; Wang, X.Y.; Jia, M.K.; Hou, C.J. Theory and application of supporting in deep soft roadways. *Chin. J. Geotech. Eng.* **2008**, *30*, 632–635.
2. Kang, H.P.; Wang, G.F.; Jiang, P.F.; Wang, J.C.; Zhang, N.; Jing, H.W.; Huang, B.X.; Yang, B.G.; Guan, X.M.; Wang, Z.G. Conception for strata control and intelligent mining technology in deep coal mines with depth more than 1000 m. *J. China Coal Soc.* **2018**, *43*, 1789–1800.
3. Kang, H.P.; Jiang, P.F.; Huang, B.X.; Guan, X.M.; Wang, Z.G.; Wu, Y.Z.; Gao, F.Q.; Yang, J.W.; Cheng, L.X.; Zheng, Y.F.; et al. Roadway strata control technology by means of bolting-modification-destressing in synergy in 1000 m deep coal mines. *J. China Coal Soc.* **2020**, *45*, 845–864.
4. Zhang, Z.F.; Kang, H.P.; Jiang, Z.Y.; Li, W.Z.; Jiang, P.F.; Cai, R.C.; Zhu, Y.T.; Wang, J. Study and application of high-pressure splitting grouting modification technology in coalmine with depth more than 1000 m. *J. China Coal Soc.* **2020**, *45*, 972–981.
5. Jing, H.W.; Meng, Q.B.; Zhu, J.F.; Meng, B.; Yu, L.Y. Theoretical and technical progress of stability control of broken rock zone of deep roadway surrounding rock. *J. Min. Saf. Eng.* **2020**, *37*, 429–442.
6. Jiang, C.Y.; Liu, Y.; Han, L.C.; Wang, C. Deformation characteristics and support technology of high-stress soft rock roadway in deep mine. *Coal Eng.* **2021**, *53*, 47–51.
7. Sun, H.L.; Zhao, J.Y.; Sun, J.Y. Study on high-pressure grouting technology with combined anchor and cable under high ground stress roadway. *Coal Sci. Technol.* **2020**, *48*, 68–73.
8. Hou, C.J. Effective approach for surrounding rock control in deep roadway. *J. China Univ. Min. Technol.* **2017**, *46*, 467–473.
9. Wang, X.Y.; Bai, J.B.; Hu, Z.C. Study on Rheological Properties of Soft Rock Roadway and Its Control Technique. *Coal Eng.* **2008**, *348*, 73–75. [CrossRef]
10. Kang, H.P. Development and prospects of support and reinforcement materials for coal mine roadways. *Coal Sci. Technol.* **2021**, *49*, 1–11.
11. Jiang, Y.; Guo, F.; Kong, H.; Lu, Y.; Sun, B.T. Current situation and development strategy of grouting material. *New Chem. Mater.* **2021**, *50*, 282–286.
12. Huang, S.J.; Zhao, G.M.; Meng, X.R.; Cheng, X.; Gu, Q.H.; Liu, G.; Zhu, S.K. Development of Cement-Based Grouting Material for Reinforcing Narrow Coal Pillars and Engineering Applications. *Processes* **2022**, *10*, 2292. [CrossRef]
13. Jiang, D.; Cheng, X.; Luan, H.; Wang, T.; Zhang, M.; Hao, R. Experimental investigation on the law of grout diffusion in fractured porous rock mass and its application. *Processes* **2018**, *6*, 191. [CrossRef]
14. Guan, X.M.; Zhong, Q.F. Research on the Performance of Micro-fine Cement-based Grouting Material and its Engineering Application. *Saf. Coal Mines* **2013**, *44*, 142–145.
15. Wang, J.F.; Zhang, H.B.; Guan, X.M. The study on micro-fine cement grouting matial applying in coal reinforcement. *J. Henan Polytech. Univ. (Nat. Sci.)* **2011**, *30*, 145–148.
16. Yu, W.J.; Zhou, M.J.; Wan, X.; Guan, Q.J. Experimental study on physical properties of superfine cement grouting material. *Front. Mater.* **2022**, *9*, 1056135. [CrossRef]
17. Guo, Y.; Zhang, B.L.; Zheng, X.G.; Zhou, W.; Wei, X.L.; Zhu, D.X. Grouting reinforcement technology of superfine cement with wind oxidation zone based on time-varying rheological parameters of slurries. *J. Min. Saf. Eng.* **2019**, *36*, 338–343.
18. Wu, A.X.; Yu, S.F.; Han, B.; Wang, Y.M.; Huang, M.Q.; Wang, Y. Optimization of mix-proportion and diffusing rule of superfine cement grouting slurry. *J. Min. Saf. Eng.* **2014**, *31*, 304–309.
19. He, F.L.; Yang, Z.Q.; Wen, Z. Optimal Studying of Grouting Reinforcement of Broken Coal Roadway Under Mining Influence. *Coal Min. Technol.* **2017**, *22*, 50–54.
20. Zhou, W.B. Application Analysis of Industrial Waste Residue Ultrafine Powder in Cement and Concrete. *Cem. Guide New Epoch* **2023**, *2*, 1–11.

21. Pitt, M.J.; Liu, H.J. Marsh funnel and and Drilling Fluid Viscosity: A New Equation for Oilfield Application. *Energy Conserv. Pet. Petrochem. Ind.* **2001**, *12*, 28–31.
22. Axelsson, M.; Gustafson, G.; Fransson, A. Stop mechanism for cementitious grouts at different water-to-cement ratios. *Tunn. Undergr. Space Technol.* **2009**, *24*, 390–397. [CrossRef]
23. Shimada, H.; Hamanaka, A.; Sasaoka, T.; Matsui, K. Behaviour of grouting material used for floor reinforcement in underground mines. *Int. J. Surf. Min. Reclam. Environ.* **2014**, *28*, 133–148. [CrossRef]