
Effects of Capsules on Compressive and Resistance of Concrete

Mingming Fu^{1, a}, Xiaoqing Gou^{1, b}, Susu Bian^{1, c}

¹Department of Chemical Engineering and Safety, Binzhou University, Binzhou 256600, China.

Zhenghemaodun@sina.com

Abstract

In view of the problem of repairing the crack in the deep mine roadway and based on a self-curing healing system, capsules are incorporated into concrete. The compressive strength and flexural strength of the sample are determined by taking the size, dosage and wall thickness of the capsules as variables. It is found that the compressive strength and flexural strength of concrete increase as the increase of capsule size and dosage. And the influence of capsule dosage on compressive strength and flexural strength of concrete is greater than that of size and wall thickness, and the concrete is more stable because of the addition of capsules. The effect and the law of capsule on the structure and performance of concrete is further clarified, which lays the foundation for the application of capsule concrete in maintaining rock mass balance in deep mining.

Keywords

Capsules; Concrete; Compressive strength; Flexural strength.

1. Introduction

1.1 Background and Significance

Coal plays an important role in social and economic development. But safety accidents caused by mine pressure should not be underestimated as the increase of mining efforts. In the past seven years, there have been 99 accidents of rock mass instability caused by roadway surrounding rock failure and it makes 371 persons die[1]. The crack in the roadway side is getting stronger under the action of mine pressure because of constantly increasing of mining depth. The stress concentration factor at the tip of the crack will also increase. With the expansion of cracks, roadway side is more prone to shear slip damage leading to the sloughing, even affecting the balance of roadway side-roof that causing a big roof accident. Therefore, it is crucial to maintain roadway side of rock mass. Analyzing shear slip of roadway side surrounding rock breaking in the shallow part can't fully satisfy roadway maintenance under high ground stress in the deep part. Deep mine roadway side support problems can be effectively solved if the crack can be found in a timely and effective manner and the expansion of the mesoscopic crack is suppressed. It also can maintain the long-term balance of anchor spray support small structure-surrounding rock balance large structure. It can not only improve safety and save a lot of roadway maintenance costs, but also has broad application prospects.

Research ideas of capsule concrete self-repairing lane crack is proposed On the basis of a "fracture generation - capsule rupture - healing agent release - healing agent curing" fracture healing system [2].

1.2 Analysis of Research Status

Capsule concrete consists of matrix material (concrete) and self-repairing (capsule) system[3]. These will affect the structure and performance of capsule concrete material. On this basis, author explains

and analyzes characteristics of self-healing repair system and research status of relationship between structure and properties of capsules in concrete.

(1) The shape and wall material of self-repairing (capsule) system. Self-repairing system (capsule) is used to encapsulate healing agent. Capsules mainly include microsphere capsules and tubular capsules [4] currently. Diameter of microsphere capsule is generally from micron to millimeter that mainly used to repair microcracks. Compared with microsphere capsules, tubular capsules have the large volume. And its healing agent has the high content that is used as healing macroscopic fissures. Macroscopic fissures are the major factor that make the roadway-roof balance state unbalanced in the deep mining of the mine. And healing macroscopic fissures is the emphasis of preventing roof accidents. However, scholars pay more attention to the mechanical properties of capsule wall materials at present. But little research has been done on the compatibility of capsule wall materials with healing agents. This problem will directly affect the service life of the capsule in concrete, so it needs to be studied in depth.

(2) Relationship between compressive and flexural properties of capsule and concrete. If capsules are put in concrete, the mechanical properties of concrete will be affected. This has strict requirements on the size, dosage and wall thickness of the capsule. The purpose of this study is to achieve the minimum impact of capsules on the compressive and flexural properties of concrete. Rule with his companies indicates the crack is difficult to heal when the crack volume exceeds the repair limit of the healing agent in the capsule [5]. Experiments done by Feng also show that less capsule loading can cause partial cracks to not be completely filled [6]. When capsules are put in concrete, it is not easy to survive during mixing if the wall of capsule is thin; and the healing agent is difficult to release and causes greater defects in the structure of the concrete if the wall of capsule is thick. This shows that the size, dosage and wall thickness of capsules have an important influence on the compressive properties and flexural strength of capsule concrete. At the same time, the capsules are irregularly distributed inside the concrete after mixing the capsule with the concrete. This distribution will also have an effect on the microstructure of the capsule concrete. And it also influences the toughness and compressive/tensile strength of concrete. Therefore, it lays the foundation for maintaining the balance of rock mass in deep mining in capsule concrete when the effect of capsule size, dosage and wall thickness on the microstructure and mechanical properties of capsule concrete are studied in depth.

2. Program Design and Results Analysis

2.1 Experimental Materials and Main Effects

Table 1. Experimental material use and proportioning table

Name	Material	Function	Proportion
Prepolymer	Urea, Formaldehyde solution Triethanolamine	Preparation of prepolymer PH regulator	Urea: Formaldehyde solution=1: 2
Core material	Epoxy resin, Acetone	Preparation of core material	Epoxy resin: Acetone =5: 1
0.5% sodium dodecyl benzene sulfonate (SDBS) solution	Sodium dodecyl benzene sulfonate (SDBS), Distilled water	Surfactant	Sodium dodecyl benzene sulfonate (SDBS): 0.5g Distilled water: 100ml
Emulsifier OP-10	Emulsifier OP-10	Emulsifier	Moderate amount
N-butane	N-butane	Defamer	Moderate amount
concrete	Plaster Quartz sand Lime Borax solvent	Cementing material aggregate Auxiliary cementing material Retarder	Plaster: Lime: Borax Solvent : Quartz Sand=3: 7: 10: 50

2.2 Capsule Preparation

2.2.1 Prepolymer Preparation

- (1) Open the water bath switch, adjust the temperature of the water bath to 60°C. and heat;
- (2) Mix urea and formaldehyde solution in a ratio of 1:2 (generally choose urea 20g, formaldehyde solution 40g), drop in triethanolamine, adjust the PH of the solution to 8~9, stir well, put in a water bath to heat, turn on the electric agitator switch, adjustment time is 40min, speed is 400rpm/min, obtain a colorless and transparent prepolymer, cool to normal temperature;

2.2.2 Core Material Preparation

Dilute epoxy resin and acetone are in a ratio of 5:1, add 3~4 drops of n-butane to deform;

2.2.3 Emulsification

- (1) 0.5% sodium dodecyl benzene sulfonate solution is configured with distilled water, add 20% dilute sulfuric acid solution to adjust PH to neutral, spare;
- (2) Take 30g prepolymer, a certain amount of emulsifier OP-10 and a certain amount of epoxy resin, (the ratio of prepolymer to core material is maintained between 1 and 1.2) mix in a three-necked flask to form an oil-in-water emulsion, put it in a water bath to heat, adjust the temperature to 50°C, adjust the electric stirrer time to 40min and the speed is 800rpm/min;

2.2.4 Acidification

Add 20% dilute sulfuric acid solution to the three-necked flask, adjust the pH of the solution in the three-necked flask to between 3 and 4, and adjust the temperature of the water bath to 60°C, adjust the electric stirrer time to 1.5h and the speed is 600rpm/min. As the reaction proceeds, the core wall resin gradually deposits on the surface of the core material;

2.2.5 Curing

Add 1g of resorcinol to the three-necked flask, adjust the electric stirrer time to 1.5h, and rotate at 400rpm/min. At this stage, the strength of the capsule wall is increasing;

2.2.6 Drying

Pour the obtained microcapsules into a beaker, add 2% sodium carbonate solution, wash with water for 3~4 times and filter, the filtered microcapsules are dispersed on an evaporating dish and dried in a vacuum drying oven at 40°C, put them in a sealed pocket and seal them after obtaining the dried microcapsules.

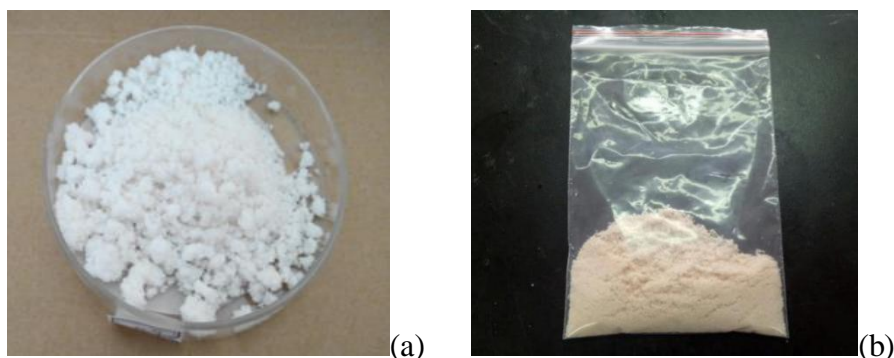


Fig 1. Microcapsule initial test sample

Observing the microcapsules with a digital microscope, the observation chart is as follows:

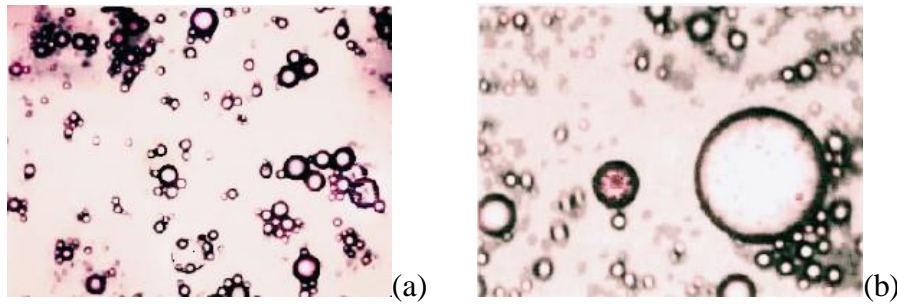


Fig 2. Microcapsule structure under the microscope

2.3 Technical Principle

(1) Based on the self-repair technology of composite cement-based materials of MF capsules: The capsule is incorporated into concrete to prepare a capsule concrete material with self-repairing function. It is for concrete structural engineering and has intelligent repair function.

(2) Moderate choice of capsule size: If the size of the capsule is too large, it will reduce the mechanical properties of the concrete; if the size of the capsule is too small, the release amount of the healing agent is small, and the investment is expensive.

(3) Appropriate amount of capsule dosage: If the dosage of the capsule is too little, the dose of the healing agent is insufficient, and the healing effect is not good; if the dosage of the capsule is too much, the mechanical properties of the material will be reduced.

(4) Crack self-healing mechanism: When the external load causes the capsule concrete to crack, the capsule at the crack is subjected to compression cracking, And under the action of capillary siphon, the healing agent flows out of the capsule to the crack, a series of flow curing reactions are carried out to seal the crack.

2.4 Capsule Concrete Preparation (5:1)

- (1) Weigh 1000g quartz sand, pour into the basin;
- (2) Weigh 140g lime and lay it on top of quartz sand;
- (3) Weigh 60g gypsum and 1% borax solvent 200g respectively, put them in the basin successively;
- (4) Stir all the contents of the basin with a shovel and make it dark gray;
- (5) Take half of the mixed material into the hopper and put in a layer microcapsule, put the remaining mixture into the hopper and mix well, and basically shaped capsule concrete is obtained;
- (6) Pack the capsule concrete in the mold, lay on the shaker for 2 minutes and fill the mold slot, demold after standing at room temperature for 24 hours. Then it is placed in a standard constant temperature and humidity curing box and taken out for 7 days.

The initial sample is shown in Fig. 3:

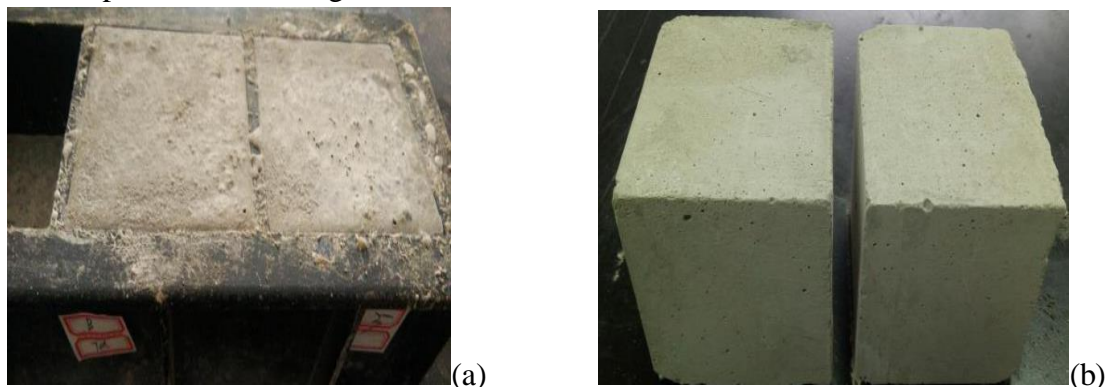


Fig 3. Formed capsule concrete Specimen

2.5 Performance Testing

2.5.1 Effect of Capsule Dosage on Mechanical Properties

Load loads with a three-point bending tester and conduct 7-day bending strength test under the conditions of the span of the pivot. Cement based composite is done pre-damage treatment by 60% σ max fixed preload, and compressive strength test is performed 7 days after self-repairing and maintenance, and record data, and compare with undamaged test piece. Substituting the obtained data into the following formula, the strength repair rate of different microcapsules on the damaged matrix can be obtained.

$$\eta = (f_{healed} / f_{initial}) \times 100\%$$

In the formula: η —the strength repair rate (%)

f_{healed} —compressive strength of specimen after self-repairing of pre-stressed damage (M Pa)

$f_{initial}$ —undamaged test piece compressive strength (M Pa)

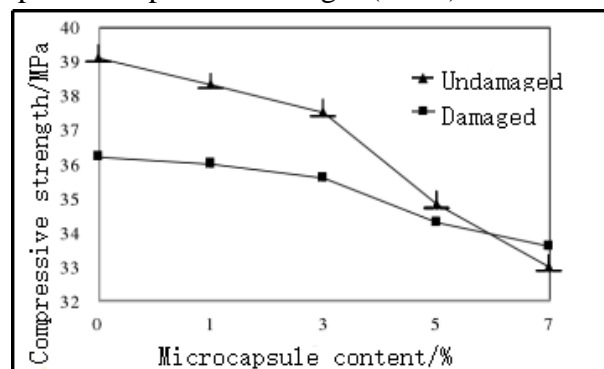


Fig 4. Damage and undamaged specimen compressive strength diagram

Fig.4 shows the original compressive strength of the undamaged test piece under different capsule dosages and the compressive strength of the self-repairing specimen under the fixed preload of 60% σ max. It can be seen from the Fig.4 that the compressive strength of the cement-based material decreases with the increase of the dosage of the capsule, but the repair rate is obviously improved. When the dosage is 5%, the strength after repair is close to the original value; even when the dosage of the capsule is 7%, the strength after repair has exceeded the original strength. It can be inferred that the capsule has a repairing effect on the crack. And when the amount of capsules is continued to increase, the repair rate will continue to increase. However, the capsule is a defect for cement-based materials. Adding capsules will reduce the strength of the concrete matrix. And if capsules are added too little, its practical value is not big. Thus, the amount of capsules should be controlled reasonably.

2.5.2 Flexural Strength and Compressive Strength

One side of the specimen is placed on the support cylinder of the bending test machine, and the long axis of the test piece is perpendicular to the support cylinder, and the load is uniformly applied to the opposite side of the prism by the loading cylinder at a rate of 50 N/s \pm 10 N/s, until the test piece is broken. At the same time, keep the two half prisms in a wet state until impressive test has finished.

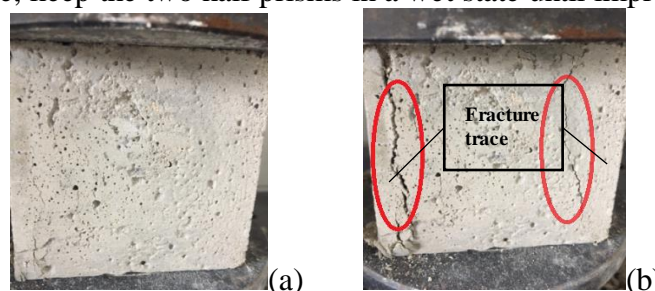


Fig 5. Capsule concrete specimen compressive strength test

The flexural strength R_f is expressed in Newtons per square millimeter (Mpa). Calculate according to equation (1):

In the formula: $R_f = \frac{1.5F_f L}{b^3}$ middle of the prism when (1) broken, N;

L--distance between the supporting cylinders, mm;

b--side length of prismatic square section, mm;

The compressive strength test is carried out on the side of the half prism by the compressive strength tester and the clamp for the compressive strength tester. The center of the half-prism cylinder and the pressure plate of the press plate should be within ± 0.5 mm, the prism body exposed to the outside of the platen is approximately 10 mm. Uniformly loading at a rate of $2400 \text{ N/s} \pm 200 \text{ N/s}$ throughout the loading process until destruction. Compressive strength R_c takes Newton's per square millimeter (Map) as a unit. Calculate according to equation (1):

$$R_c = \frac{F_c}{A} \tag{2}$$

In the formula: F_c --maximum load at damage, N;

A--area of the pressed part, $\text{mm}^2(40\text{mm} \times 40\text{mm} = 1600\text{mm}^2)$

The three variables of the amount, length and wall thickness are used as the three factors of the orthogonal experiment, and then three levels are selected from each factor. The L9 (34) orthogonal test table is used to test the compressive and flexural strength [7]. As shown in Table 2:

Table 2. Orthogonal test results

Sample serial number	content %	lengthmm	wall thickness mm	Flexural strength Mpa	Compressive strength Mpa
1	1	20	0.6	0.79	2.60
2	1	30	1.0	0.81	2.58
3	1	40	1.4	0.84	2.72
4	3	20	1.0	0.84	2.55
5	3	30	1.4	0.86	2.58
6	3	40	0.6	0.93	2.62
7	5	20	1.4	0.74	2.45
8	5	30	0.6	0.82	2.56
9	5	40	1.0	0.85	2.62
Flexural strength Mpa	K1	0.81	0.81	0.85	
	K2	0.88	0.84	0.83	
	K3	0.80	0.86	0.81	
	R	0.08	0.05	0.04	
Compressive strength Mpa	K1	2.63	2.53	2.59	
	K2	2.58	2.57	2.58	
	K3	2.54	2.65	2.58	
	R	0.09	0.12	0.01	

In the table, K1, K2, K3 respectively represent average of three factors under three indicators, including content (%), length (mm), wall thickness (mm). R represents the difference between the maximum and minimum values of K under each indicator. It can be seen from the orthogonal table that the compression and flexural strength are optimal when the dosage is 5%, the length is 40mm, and the wall thickness is 1.4mm. And when the amount is more than 5%, the strength of the concrete is greater

than the original strength. This proves that the healing agent in the capsule has a good function of repairing cracks.

3. Conclusion and Outlook

The effect of capsules on the compressive and flexural properties of concrete is closely related to its size, dosage and wall thickness. In this study:

(1) When the capsule content is 5%, the strength after repair is close to the original value; When the dosage of the capsule is 7%, the strength after repair has exceeded the original strength.

(2) When the capsule is 20mm long and the wall thickness is 0.6mm, the capsule has broken.

(3) Through the recovery test of mechanical properties, the prepared capsule concrete has the characteristics of mechanical properties, and achieves the expected healing target to a certain extent.

The results of this study lay the foundation for the application of capsule concrete materials in repairing deep roadway cracks. Since the capsule is a defect for concrete, the influence of the capsule composition on the compressive and flexural properties of the concrete needs further research and discussion.

Acknowledgements

Project Number Of Shandong Province Natural Science Foundation: ZR2017PEE024.

The Doctoral Scientific Research Foundation Number: 2017Y09 SRTP Number: 234.

References

- [1] State Administration of Work Safety, State Administration of Work Safety, Government Website Accident Inquiry System[z].2010-2017.
- [2] Zhang SX, Wang DM, et al.: Survey of research on self-repairing system of cement-based microcapsules [J].Commodity concrete, 2015 (04):25-28.
- [3] Dong B, Wang Y, Fang G, Han N, Xing F, Lu Y. Smart releasing behavior of a chemical self-healing microcapsule in the stimulated concrete pore solution. Cement & Concrete Composites, 2015, (56): 46-50.
- [4] Bengzon Tan NP, Keung LH, Choi WH, Lam WC, Leung HN. Silica-based self-healing microcapsules for self-repair in concrete. Journal of Applied Polymer Science, 2016, (12). DOI: 10.1002/app.43090.
- [5] Rule JD, Sottos NR, and White S. Effect of microcapsule size in the performance of self-healing polymers. Polymer, 2007, (48): 3520-3529.
- [6] Feng X, Zhao Nets. Self-healing mechanism of a novel cementations composite using microcapsules. In pricedings of international conference on durability of concrete structures, Hangzhou, China, 2008.
- [7] GB/T17671-1999 Cement sand strength test method (ISO method) [S].