

Article

Experimental Study on the Physical and Mechanical Properties of Cemented Gangue Backfill under Acid Mine Water Erosion

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Abstract: Ensuring the structural safety of cemented gangue backfill (CGB) is crucial for safe mining operations. However, the complex mine water environment makes it susceptible to erosion by chemical ions, which have a significant time dependency. In this study, we evaluated the appearance, mass change, and unconfined compressive strength (UCS) of CGB during different chemical erosion times. We also determined the effect of chemical ion erosion time on the stress threshold for crack initiation and development in the specimens using acoustic emission (AE). Additionally, we examined the chemical erosion mechanism of CGB by scanning electron microscopy (SEM). Our results showed that as the erosion time increased, the CGB exhibited a decrease in brittleness and an increase in plasticity. During the first 60 days of CGB, the internal micropores and microfractures of the CGB were filled due to the hydration reaction and SO_4^{2-} intrusion, resulting in increases in the UCS and the mass of the CGB. However, as the erosion time continued, H^+ and SO_4^{2-} intruded into the interior of the CGB, causing the erosion products of the CGB to expand in volume, leading to a decrease in the strength of the CGB. Our analysis of the stress thresholds for microcrack development and macrocracks initiation in the CGB showed an increase followed by a decline with time. After 60 days of immersion, the stress threshold for microcrack initiation and macrocrack extension increased by 20% and 6%, respectively. However, as the immersion time increased to 150 days, the stress threshold for microcrack initiation and macrocrack extension decreased by 56% and 16%, respectively. Therefore, the design of CGB safety needs to consider the long-term effects of chemical attacks on CGB.

Keywords: chemical erosion; mechanical properties; acoustic emission; stress threshold; erosion mechanism



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1. Introduction

Underground mining can create large underground cavities and leave large amounts of mine waste on the surface. Without proper support, an underground chamber can easily lead to surface subsidence. The disposal of mine wastes on the Earth's surface can cause problems such as acid mine drainage [1]. In the last few decades, CGB has become one of the effective methods to solve the problem of gangue solid waste discharge and surface subsidence during mine production [2–4]. The hydrogeological structure of China's coal-producing areas is complex, and the coal-mining process is often accompanied by the generation of large quantities of mine water [5]. Due to the high number of sulfide minerals (usually pyrite in coal seams), sulfide minerals are susceptible to oxidation when exposed to water and oxygen during coal mining, making the mine water acidic [6]. According to statistics, the mine water from many coal mines worldwide commonly contains Ca^{2+} , Mg^{2+} , HCO_3^- , SO_4^{2-} , and heavy metal ions, presenting a low pH value [7]. In China, the pH of the mine water in coal mines is generally in the range of 2.5 to 5.8, and the pH of the

mine water in certain areas can even reach 2.0, making the mine water rich in hydrogen ions. As a cementitious material, CGB is internally susceptible to erosion by chemical ions when it is in an acidic mine water environment, which affects the integrity and strength of the material and fails to achieve the purpose of controlling surface settlement in the long term [8]. Therefore, it is necessary to study the effect of chemical erosion time on the mechanical behavior and fracture process of CGB, as well as the damage mechanism of CGB under chemical erosion.

Sulfide minerals in contact with water and oxygen are prone to chemical reactions and oxidation to sulfate (SO_4^{2-}) and hydrogen ions (H^+), which expose the CGB to external chemical ions [9,10]. On the one hand, the hydrogen ions formed will destroy the hydration products of cement; on the other hand, the sulfate ions will react chemically with the hydration products to produce ettringite, which leads to a more complex change in the strength of the CGB [11]. A lot of research work is currently devoted to the study of sulfate changes in the physical and mechanical properties of the CGB [12–14]. It has demonstrated that the initial sulfate content in the interior of the CGB significantly reduces its long-term UCS, implying that one of the factors contributing to the structural deterioration of the CGB is an internal sulfate attack. Guo et al. [15] studied the diffusion process of sulfate ions in cemented gangue backfill material. The results showed that the diffusion rate of sulfate ions was affected by the internal pore structure, and the changes in the internal pore structure were related to the reaction products at different times. Dong et al. [16] showed that sulfide erosion decreases the strength and structural stability of cementitious materials. The most significant decrease in strength was observed in the CGB specimens with the highest sulfur content. Wang et al. [10] investigated the effect of exogenous sulfate erosion on the mechanical behavior and properties of CGB, and the results showed that the sulfate concentration is a hydration product formation important factor which directly affects the macroscopic behavior and volume of CGB before and after damage. The studies mentioned above indicate that chemical ion erosion impacts the microstructure of CGB, which leads to a change in the mechanical response. Therefore, it is crucial to investigate how chemical erosion affects the stress threshold of CGB crack development. However, these studies focus more on examining the changes in the macroscopic mechanical properties of CGB due to chemical erosion rather than on studying its fracture behavior.

The acoustic emission (AE) signal can reflect both the material's internal changes and the material's mechanical response under loading [17]. The acoustic emission technique is helpful in analyzing the internal fracture process of chemically eroded CGB under loading. Zhao et al. [18] studied the acoustic emission characteristics of backfill with different ash-to-sand ratios under different loading modes. They found that the split specimens' acoustic emission activity patterns were the same as those under uniaxial compression. During the splitting test, the filled specimens did not exhibit any acoustic emission activity and behaved calmly before the peak stress period. When the stress reached the peak, the acoustic emission activity level became abnormally high and reached the maximum value. Cao et al. [19] investigated the acoustic emission characteristics of colluvial ending sands under different loading rates. They found that the distribution of ringing counts during the loading process had a regular peak spacing. The cumulative number of ringing counts also showed a "stepwise" growth trend and eventually stabilized. Wu et al. [20] studied the effect of aggregate gradation on the acoustic properties of compressed cemented mounds. They found that the acoustic emission signal activity was positively correlated with the Talbot index of cemented mound aggregates. These findings contribute to a better understanding of the AE characteristics of CGB during loading. However, most previous studies have focused on the acoustic emission characteristics of filled bodies with different material ratios or loading methods. The acoustic emission characteristics of CGB loaded by chemical ion erosion have not been thoroughly investigated.

Considering the importance of the effect of chemical erosion on the performance of cemented gangue backfill, this paper attempts to investigate the acoustic, mechanical, and physical changes in CGB due to the erosion time of chemical ions. First, fresh CGB was

prepared and processed into standard samples. The samples were immersed in the same pH solution for different times using the laboratory accelerated corrosion test method. Then, we conducted a uniaxial compression test, recorded the acoustic emission signals, and investigated the effect of chemical ion erosion on the damage characteristics of CGB during loading using acoustic emission signals. Finally, the microstructure and chemical composition changes in the eroded backfill were analyzed by SEM.

2. Material and Methods

2.1. Preparation of Material

Considering the main distribution area of acid mine water in China, the gangue used was taken from a mining site in Jining, Shandong Province, the fly ash was taken from a neighboring power plant, and the cement was ordinary silicate cement P.O 42.5. The Talbot gradation index of the gangue particles, N , was taken to be 0.6. Combined with the relevant research by scholars [21], the minimum diameter of the cylindrical specimen must be more than five times smaller than the largest aggregate particle size. The gangue samples collected from the mine site were crushed into finer particles, and then graded into six different particle size intervals: (0.0–0.5) mm, (0.5–1.5) mm, (1.5–4.0) mm, (4.0–6.0) mm, (6.0–8.0) mm, and (8.0–10.0) mm. The mass fraction of gangue for each particle size interval was obtained using the Talbot gradation formula. The mass fraction of each interval of particle size is shown in Table 1.

Table 1. Mass fraction of gangue particles in each particle size range.

Particle size range (mm)	0~0.5	0.5~1.5	1.5~4.0	4.0~6.0	6.0~8.0	8.0~10.0
Mass fraction (%)	16.57	15.47	25.67	15.89	13.87	12.53

The chemical compositions of gangue, fly ash, and cement used for the experiment were tested, and the test results of the mass fraction of their main chemical compositions are shown in Table 2. The specimens were made into cylindrical specimens of $\phi 50 \times 100$ mm with a height-to-diameter ratio equal to 2 according to the International Society for Rock Mechanics' (ISRM) recommended methods and standards. The total water/cement ratio of the specimen was taken as 0.75, and the material proportions of the specimen were 61.2 g of cement, 6.8 g of fly ash, and 340 g of gangue.

Table 2. The physical parameters of selected samples.

Major Element	Gangue (%)	Cement (%)	Fly Ash (%)
SiO ₂	55.7	20.41	47.45
Al ₂ O ₃	19.88	6.01	26.28
CaO	1.58	64.95	7.11
MgO	2.00	1.27	0.85
Fe ₂ O ₃	3.92	3.50	12.21
TiO ₂	1.63	0.18	0.41
SO ₃	0.20	2.17	0.73
K ₂ O	0.12	0.47	0.95
LOI	14.92	1.88	3.93

2.2. Experimental Methods

This test adopted a laboratory accelerated erosion program; the erosion medium selected sulfuric acid solution. First, H₂SO₄ solution with a concentration of 1 mol/L was used to prepare, and the final pH value was 3.0. The cemented gangue backfill specimens were immersed in the same pH value sulfuric acid solution in a container for 28 d. The erosion times of cemented gangue backfill were set as 30 d, 60 d, 90 d, 120 d, and 150 d, respectively. Excluding the initial time (0 d), three samples were tested at each erosion time, totalling fifteen samples tested. The aqueous sulfuric acid solution was reformulated and

replaced every three days during the test in order to keep the CGB specimens immersed in a relatively constant pH environment. Then, the CGB specimens with different erosion times in the aqueous sulfuric acid solution were taken out in time, wiped off the liquid on the surface of the specimens, and put into the drying oven for 12 h (Figure 1). In the next step, an MTS816 rock mechanics testing machine was used to carry out the uniaxial compression test of the chemically eroded CGB specimens, and the acoustic emission signal monitoring was carried out during the uniaxial compression test [22]. The AE signals generated during experiment were collected by the AE sensor (R6 α) provided by the AEWIn acoustic emission system manufactured in the United States. The operating frequency of this sensor is 35–100 kHz. It is important to mention that before each acoustic emission test, it is necessary to detect the noise level of the testing environment to set an appropriate acoustic emission threshold. Finally, the immersed specimens were microscopically scanned with an SEM electron microscope [23].

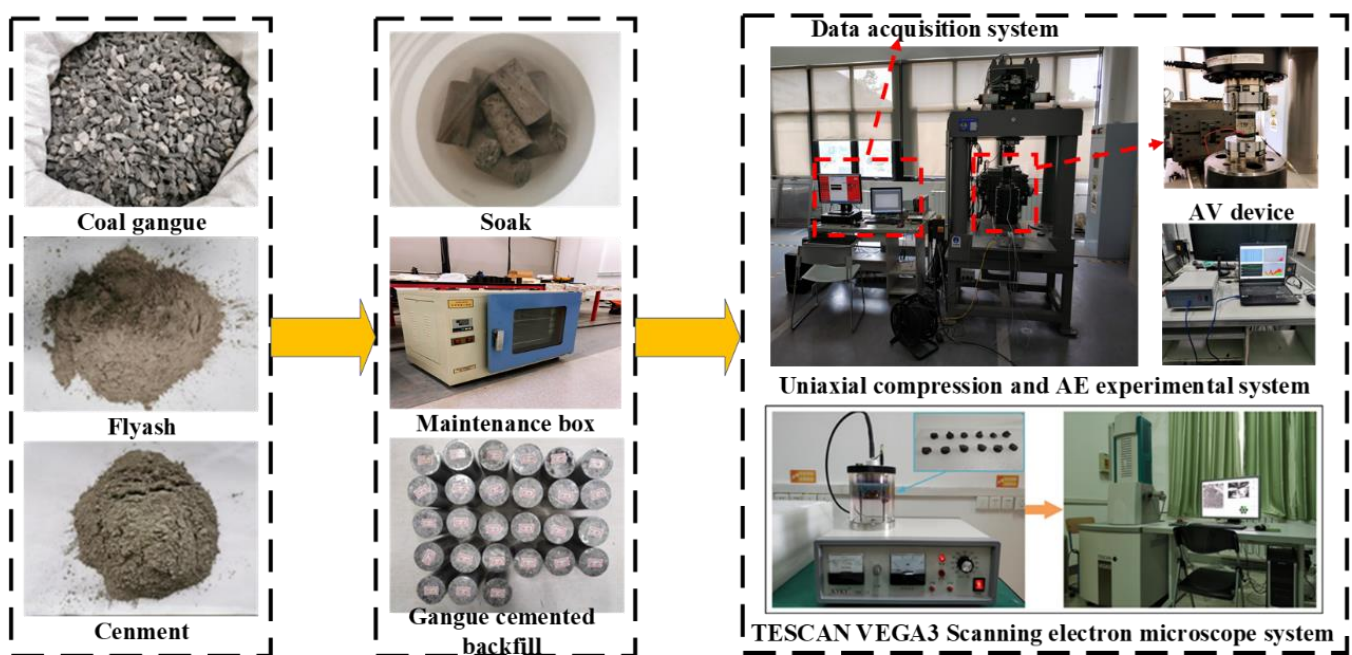


Figure 1. Experimental procedure and equipment.

3. Results and Discussion

3.1. Evolution of the Mass and Appearance

As the chemical ion erosion time increases, the appearance of CGB specimens at different periods is shown in Figure 2. At the early stage of erosion, the surface morphology changes are not noticeable; the shape is intact, and only some tiny pits appear. With the extension of the erosion time, the surface started to float slightly; when the erosion time increased to 120 days, CGB surface white crystals precipitated. At the same time, in the CGB surface layer dissolution phenomenon, the aggregate particles appear to be partially exposed, and the filling body from the outside to the inside of the whole became “fluffy.” After 150 days of erosion, the surface of the filling body turned yellow, and the surface layer was partially detached, with corrosion products attached, and numerous tiny holes and microcracks appeared.

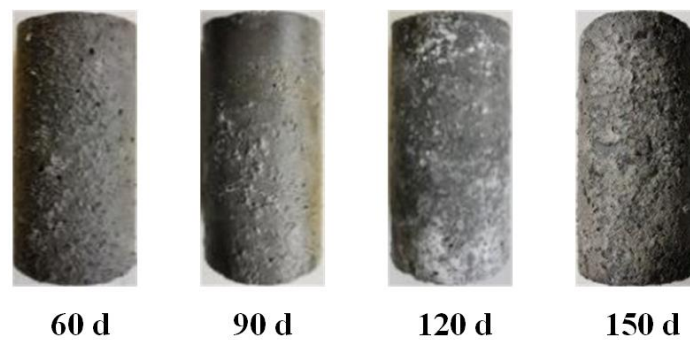


Figure 2. The apparent morphology of the sample after soaking at different times.

The recorded mass change in CGB under different erosion times, and the relevant data were shown in Figure 3. It can be found that with the increase in the erosion time, the mass of the CGB specimens immersed in the acidic aqueous solution did not decrease immediately at the beginning of erosion, but showed a trend of increasing first, and then decreasing. When the time of erosion reached 60 days, the mass of the specimen increased by 1.6% compared with that of the initial one, and then it began to decrease gradually. When the time of erosion reached 150 days, the rate of change in specimen mass was -0.5% , indicating that the specimen mass was lower than the initial specimen mass. Combined with the specimen's appearance in Figure 2, the ions in the erosion solution entered the specimen through the pores on the surface of the specimen to undergo a chemical reaction. The generated insoluble substances adhered to the inside of the pores, leading to a decrease in the overall porosity of the specimen, making the total mass increase. As erosion time was prolonged, excess insoluble material accumulated, leading to the expansion of microcracks within the specimen and the formation of small cracks. More erosion ions from the solution entered the interior of the CGB, and eventually, some of the products dissolve, leading to a decrease in the total mass.

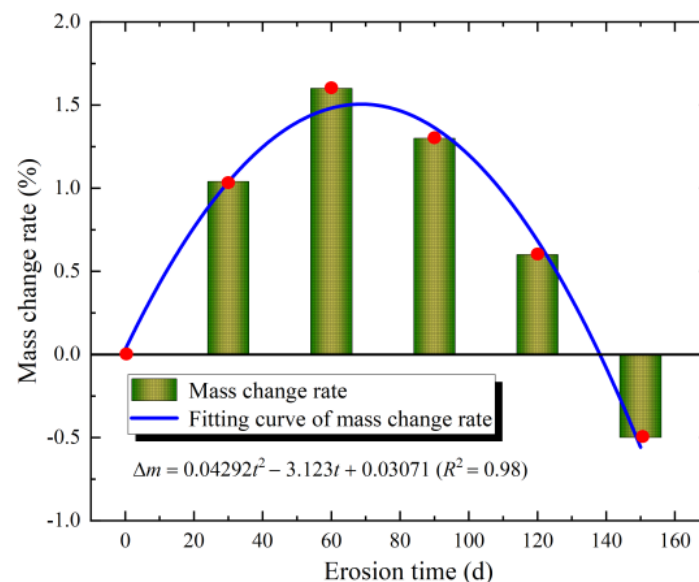


Figure 3. Mass change in CGB after chemical erosion.

3.2. Evolution of the UCS

A uniaxial compression test was carried out on CGB after chemical corrosion. A typical stress–strain curve of CGB after different times of erosion was obtained, as shown in Figure 4a. It can be seen from the figure that the shape of the curve shows different trends with the increase in the CGB erosion time. When the erosion time reached 60 days, the uniaxial compressive strength of the specimen increased by 16% compared with that of the

initial specimen. When the erosion time increased to 150 days, the uniaxial compressive strength decreased by 8% from the initial specimen and by 20% from that after 60 days. As the erosion time increases, the initial compressive and yield stages of the stress–strain curves are lengthened, while the elastic phase is shortened. The peak strain of the CGB increases, the ratio of peak stress to strain decreases with the erosion time, and the curve segment from the beginning of the loading to just before the peak stress flattens out. The reason for this is the increase in erosion time and a large number of SO_4^{2-} and H^+ invaded, resulting in the formation of more micropores on both the surface and the interior of CGB so that the original pores of the specimen are enlarged and the chemical erosion reaction zone is enlarged. Macroscopically, it is manifested in the reduction in brittleness, the enhancement of plasticity, and the increase in the deformation of CGB when subjected to a load. It can be seen that the erosion of acidic aqueous solution has a significant effect on the mechanical properties of CGB, resulting in changes in its mechanical response to external loads.

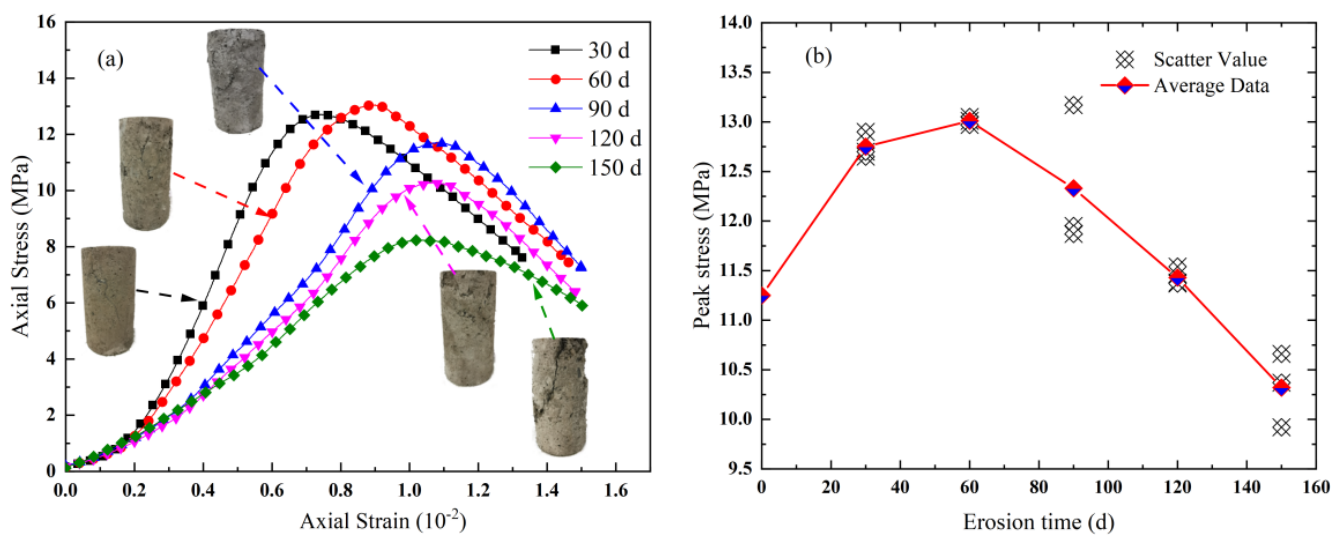


Figure 4. (a) Typical stress-strain curve and peak stress variation in CGB after chemical erosion. (b) Variation in CGB peak stress with erosion time.

3.3. Effects on the AE and Failure Behavior

The deterioration caused by chemical erosion makes the fracture processes of CGB very complex. Therefore, it is crucial to understand the fracture behavior of CGB during loading. The acoustic emission data can be used to study the spatial evolution of deformation and fracture in CGBs during loading, making it an effective method for damage assessment [24]. The raw acoustic emission waveforms can be analyzed for various parameters, such as the peak counts, energy, and entropy, to determine the fracture initiation point and identify the damage stage [25]. Xu et al. [26] found a specific relationship between the acoustic emission signal and the stress–strain behavior of the material during loading. Therefore, in this study, the changes in the stress threshold of CGB during their damage process after chemical erosion were explored with acoustic emission technology, and the changes in the acoustic emission signals of the CGB samples with different erosion times were analyzed and evaluated. Ultimately, these results are related to the damages caused by chemical erosion on the CGB.

3.3.1. Acoustic Emission Behavior

The AE and cumulative AE responses over time of the samples during the loading process are shown in Figure 5. The changes in the acoustic emission counts at different chemical erosion times can be broadly categorized into initial, calm, and active phases. The figure shows that when the immersion time is less than 60 days, the initial and calm stages

of the specimens are prolonged compared to those of the fresh samples. The trend of the acoustic emission counts change in this process is relatively smooth, while the active phase is compressed, and the acoustic emission counts change faster in this phase. The result suggests that after short-term immersion, the chemical ions did not lead to the deterioration of the internal structure and the creation of pores. Instead, the internal pores and localized microscopic defects may have been filled, strengthening the internal structure. The primary source of the acoustic emission signal may be the result of inter-particle friction during the closure of the initial microcracks inside the CGB. When the initial crack closes, the elastic energy in the specimen begins to accumulate. It continues until the energy or stress required for crack extension is reached, and the specimen starts to break down. The acoustic emission counts in the initial stage showed a positive correlation with increased chemical erosion time when the chemical erosion time was greater than 60 days. During this stage, the chemical erosion action may have caused the deterioration of the internal structure of the specimen or the generation of microcracks, which led to the enhancement of the changes in the acoustic emission counts. With the increase in the chemical erosion time, the density of the acoustic emission counts in the calm period also increases relatively, and more mutation points appear before the destruction of the specimen. In this process, a portion of the energy was released through a stress waveform, resulting in a sharp increase in AE counts. The phenomenon indicates that the specimen is about to enter the unstable crack development stage and gradually reaches the peak stress point to produce damage. In addition, the duration of the active phase is influenced by chemical attacks and tends to increase. Therefore, changes in the acoustic emission behavior caused by the temporal effects of chemical erosion should be considered in engineering designs.

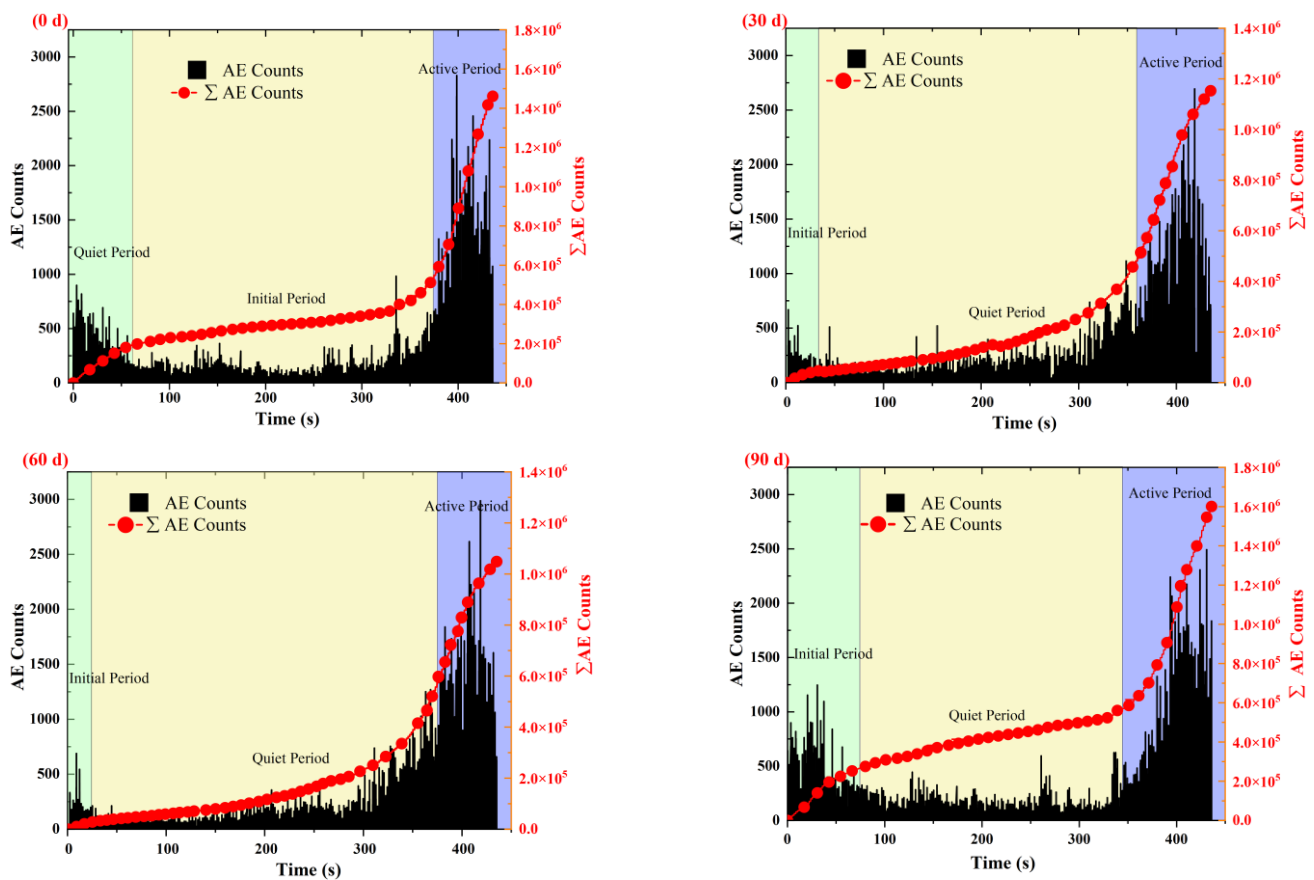


Figure 5. Cont.

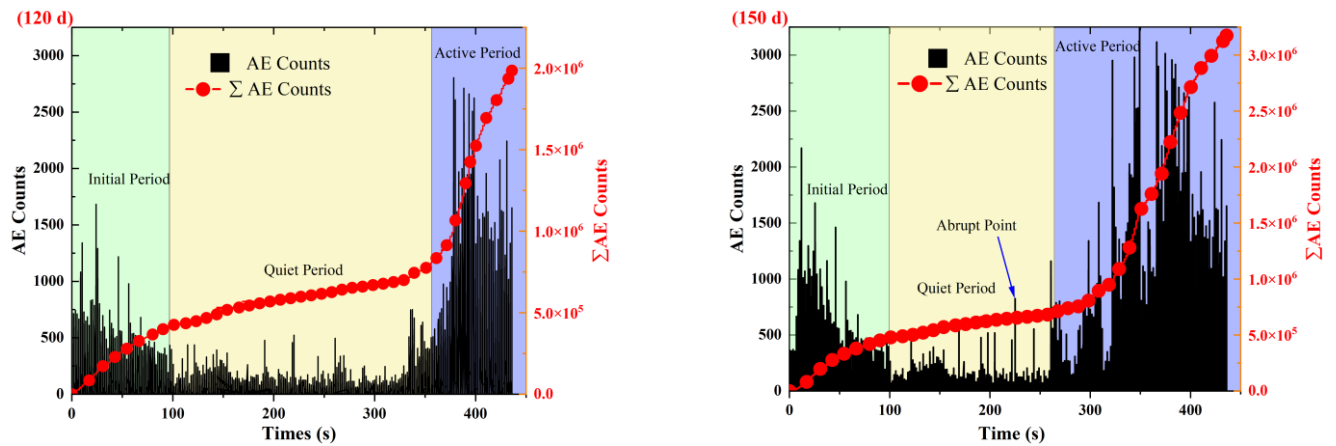


Figure 5. The relationship between AE count and cumulative AE count of samples with different erosion times under uniaxial compression.

3.3.2. Acoustic Behavior with Stress

Figure 6 shows the relationship between AE behavior and the stress of samples with different chemical erosion times under uniaxial compression conditions. According to the literature [27], rocks show four stages of changes in acoustic emissivity and slope of stress curves: initial crack closure, new microcracks sprouting, microcracks expanding and merging, the emergence of macroscopic cracks, and finally, destruction. In our study, except for Figure 6d, where the slopes showed significant changes, the slopes for the rest of the erosion time showed fewer changes. This may be because the material we used was artificially made and already contained a cementitious matrix chemically attacked by sulfate ions and hydrogen ions, and the microcracks had already appeared before loading. This made the slopes more similar in the second and third stages, and the slope change was insignificant. For a better discussion, we divided the whole into four stages using simple slope calculations. As seen in Figure 6, the AE–stress relationship curves of the specimens show a clear demarcation and keep changing, indicating that the difference in the chemical ion erosion time significantly impacts the mechanical response of the CGB to the external load. The results show that the deformation behaviors of the specimens under loading change with the chemical ion erosion time. It is important to note that visible damage refers to the extension of macroscopic cracks. When the erosion time is less than 60 days (Figure 6b,c), the microcrack initiation stage (II) of the specimen is prolonged, and the crack damage zone (IV) is more compressed compared to that of the initial sample (Figure 6a). These results indicate that chemical erosion plays a positive role in the initial immersion stage on the mechanical response of CGB to external loading. With the increase in the chemical ion erosion time (Figure 6d–f), the crack initiation stage (II) of the specimen decreases, and the macroscopic crack damage zone (IV) becomes wider. It indicates that the deformation of the specimen under less stress after entering the damage stage is quickly transformed from microcrack closure to microcrack extension, the macroscopic crack damage zone is enlarged, and the acoustic emissivity rises sharply. The results reflect that for a longer chemical erosion time, the internal structure of CGB deteriorates, and its mechanical response to external load becomes unstable. The degree of chemical ion erosion significantly affects the generation and subsequent extension of macroscopic cracks. Therefore, these results demonstrate that the chemical ion erosion time predominantly affects the acoustic emission behavior of the specimens under uniaxial compressive loading.

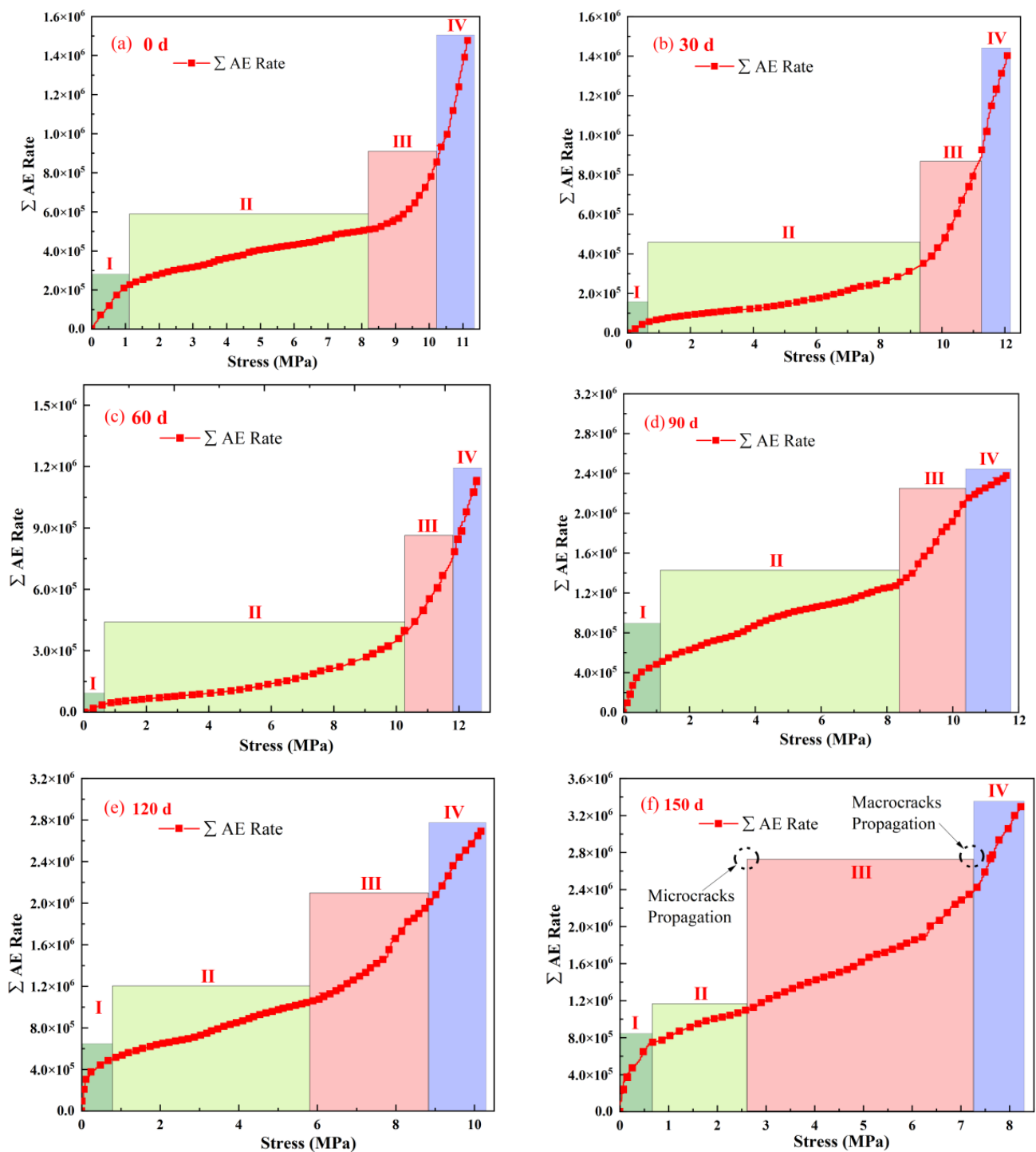


Figure 6. Acoustic emission behavior as a function of stress for specimens with different erosion times under uniaxial compression conditions. I—initial crack closure; II—new microcracks sprouting; III—microcracks expanding and merging; IV—the emergence of macroscopic cracks.

The stress threshold determines a specimen's occurrence, coalescence, and the extension of microcracks [28]. This section explores the effect of chemical attack time on the stress thresholds for each deformation stage of the CGB. Figure 6 shows the stress thresholds at each displacement point during loading. The stress thresholds are normalized by the peak stress to quantitatively analyze the changes after different erosion times, as shown in Figure 7. As observed in the figure, the stress thresholds for microcrack extension and macrocrack initiation increase, and then decrease with the increase in the chemical

ion erosion time. For instance, the normalized stress threshold for microcrack extension increases from 0.71 to 0.85, and the normalized stress threshold for macrocrack initiation increases from 0.9 to 0.96 after 60 days of erosion. However, after 150 days of erosion, the normalized stress threshold for microcrack extension decreases to 0.31, and that for macrocrack initiation drops to 0.76. These phenomena imply that chemical ion erosion significantly affects the microcrack extension and macrocrack initiation of CGB. The chemical ion erosion has a strong time-related dependence on the microcrack extension and macrocrack initiation of CGB. Even though the initial stage of erosion shows an increase in strength, microcrack extension occurs in the CGB at later stages under a low-stress input as the erosion time increases, leading to a decrease in the overall load-bearing capacity and strength. The strength of the CGB is mainly composed of the gangue aggregate particle strength, the strength of the cement hydration reaction products, and the bond interface strength gangue aggregate-hydration reaction products between the gangue and hydration reaction products. The bond interface of the aggregate-hydration reaction products was weakened in the late stage of erosion. These results demonstrate that chemical erosion time substantially affects the fracture behavior of the CGB.

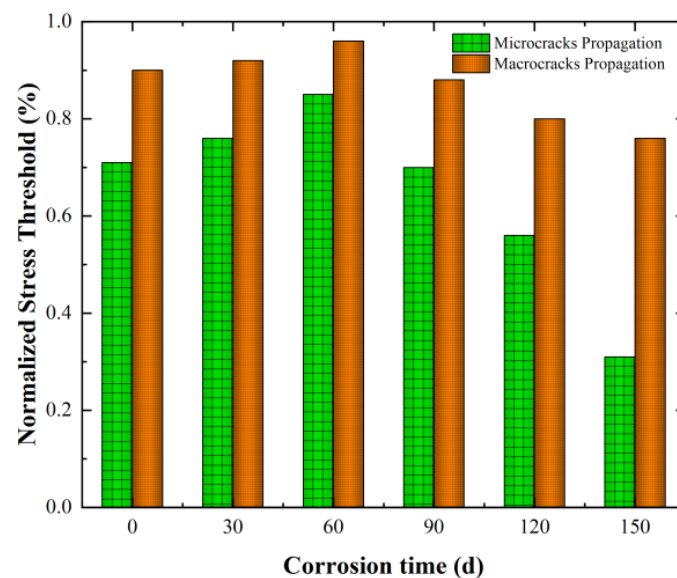


Figure 7. Acoustic properties of CGB after different erosion times.

4. Mechanism Discussion

After conducting the experiments and analyzing the results, SEM was used to observe the microstructure of CGB. This analysis helped to understand the erosion mechanism of CGB in acidic solutions. To make it easier to discuss, the reaction mechanism is divided into the following four parts.

4.1. Hydration Reaction

It is well known that CGB is strong before erosion mainly because of its internal hydration reaction (Equations (1) and (2)). The products of the hydration reaction are ettringite, calcium silicate hydrates (C-S-H), and portlandite precipitation (Figure 8a–c). Since the gangue aggregate is strong, the strength of gangue cemented filler mainly depends on the hydration products and bonding interface strength. The C-S-H structure is dense and reticulate, with a distribution of gel and capillary pores, which results in a very high specific surface. The gangue aggregate particles wrapped with C-S-H form a solid structure, which is helpful in resisting damage behavior under external loads. This solid structure is favorable to jointly resisting destructive behavior under the action of external loads [29].



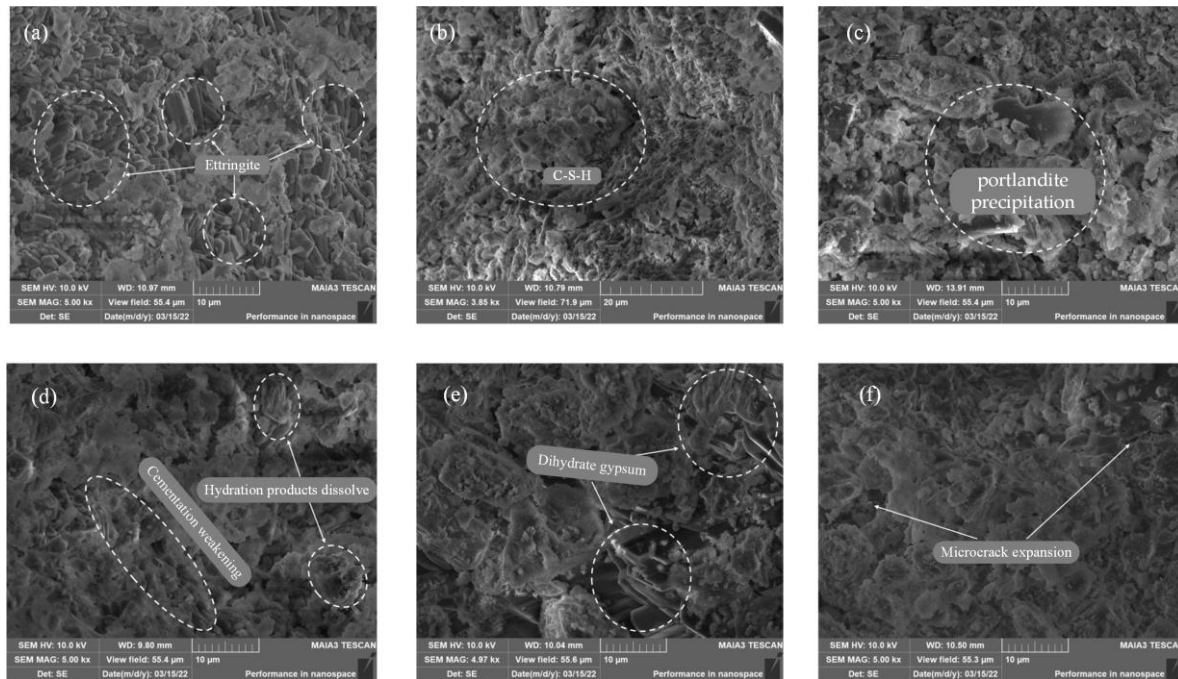
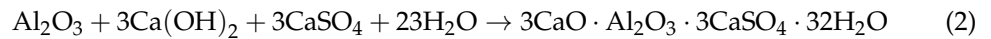
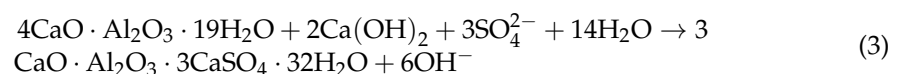


Figure 8. SEM micrograph of CGB under chemical erosion. (a–c) represent the hydration products; (d) represents the decomposition of hydration products; (e) represents gypsum precipitation; (f) represents microcrack expansion.

4.2. Formation of Ettringite

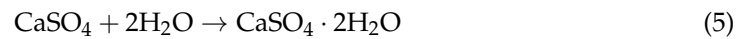
The initial formation of ettringite results from the hydration reaction in cementitious materials like cement. This reaction provides some strength to the specimen during the initial curing stage. However, as SO_4^{2-} ions diffuse/or absorb in the interconnected pores reacting with existing hydration products, the equilibrium state of the pore solution with the minerals in the CGB is disrupted, and monocarboaluminate reacts with sulfate to form ettringite in the presence of calcium hydroxide and water [30–32]. The formation of ettringite leads to a local decrease in porosity [33], which results in a denser internal structure of the CGB and a corresponding increase in mass, leading to a short-term increase in the strength of the CGB. This explains the decrease in the acoustic emission rate in the early part of the immersion. However, with the increase in the erosion time, the volume of ettringite exceeds the pore space, exerting significant expansion pressure on the microstructure and causing cracking [34]. Due to the microcracking-induced damage to the microstructure, a decreasing threshold of microcrack expansion can be seen in the acoustic emission experiments on CGB immersed for 120 d and 150 d. The specific reaction equation is as follows:



4.3. Formation of Gypsum

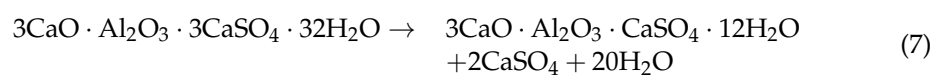
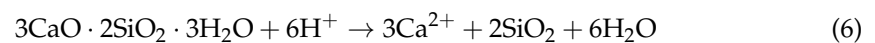
A sulfate attack can lead to significant mechanical damage (cracking), related mainly to differential internal expansions, caused, according to the literature [35,36], primarily by ettringite formation and secondly by gypsum. As sulfate ions continue to intrude, ettringite is accompanied by gypsum precipitation. Gypsum accumulation causes volumetric expansion, leading to the formation of microcracks under expansion stress (Figure 8e), which facilitates the rate of ions in the solution into the material, increasing the contact area between the ions and the material and allowing more erosion products to be produced [37].

Additionally, the production of gypsum depletes $\text{Ca}(\text{OH})_2$. It reduces the alkalinity inside the specimen, leading to the decomposition of hydration products (Figure 8d), resulting in a loss of strength and the durability of the CGB. This process further exacerbates the destruction of the CGB. The reaction equation is as follows:



4.4. Calcium Leaching

The weakening effect of calcium leaching, which involves the decomposition of ettringite and C-S-H, is considered a crucial component of the sulfate erosion mechanism. On the other hand, leaching phenomena in acidic solutions mainly cause the dissolution of portlandite and the decalcification of C-S-H [38] (Equations (6) and (7)). Dissolving the minerals increases the material's porosity and reduces its mechanical properties [39–41]. The results of Planel's study show that there are two characteristic layers in which the calcium-leached portion of the material exists and extends from the outer surface: predominantly, ettringite (zone 1) and ettringite, gypsum, and portlandite (zone 2) [42]. Since sulfuric acid solutions were used in this study, the hydrogen ions may exacerbate calcium leaching caused by a sulfate ion attack. Other studies have shown that ettringite dissolves into gypsum and aluminum sulfate when subjected to pH values below approximately 10.5 [43]. Therefore, when the surface layer of the CGB was corroded, some of the ettringite exposed to the hydrogen ions was corroded and dissolved, leading to a decrease in the mass of the CGB. The re-exposure of the pore space filled initially by ettringite due to this process changes the pore structure, which can significantly affect the specimen's permeability [44]. Under the double attack of sulfate ions and hydrogen ions, the structure and bonding interface strength of CGB hydration products are weakened, leading to a reduction in the macroscopic mechanical strength (Figure 8f). The results of UCS and acoustic emission confirmed this process.



The strength change in the CGB under chemical ion erosion can be divided into two main stages. In the first stage, the strength of the CGB increases due to the hydration reaction and the filling and compacting effect of ettringite when the alkaline environment is still dominant inside the specimen. However, as the erosion time increases, the double invasion of hydrogen and sulfate ions destroys the alkaline environment. As a result, ettringite loses its survival environment and decomposes rapidly, destroying the filler specimen's internal crystal structure in a relatively short time. Compared to the strength deterioration effect caused by acid corrosion, the hydration reaction of the cementitious materials is less strong. The strength of the filled specimen reduces, and the rate of strength deterioration accelerates with time. Additionally, the expansion stress generated by gypsum water absorption leads to microcracks formation, further exacerbating the erosion of internal ions. During load-bearing compression, the CGB structure and bonding interface become damaged, which weakens the cementation. As a result, the gangue cemented filling body is more prone to microcracks under the action of stress, especially along the interface transition zone where microfractures occur.

5. Conclusions

The physical and mechanical properties of CGB are heavily impacted by long-term exposure to acidic mine water, and this impact is time-dependent. This study investigates the changes in acoustic emission characteristics and mechanical properties of the CGB, as well as the evolution of stress threshold for crack sprouting under different exposure times.

Based on microscopic tests, this paper also analyzes the deterioration mechanism of the CGB after chemical ion erosion, and the main conclusions drawn are as follows:

1. Chemical erosion damages the apparent structure of the CGB, leading to an increase, and then decrease in its mass and UCS over time. As the erosion time increases, the initial compression stage and yield stage of the CGB stress–strain curve are prolonged, and the elastic stage is shortened. This leads to a reduction in CGB’s compression brittleness and the enhancement of its plasticity.
2. The acoustic behavior of the CGB when subjected to uniaxial compression demonstrates the impact of acidic chemical erosion on the AE–stress relationship. During the initial stages of chemical erosion, the swelling products produced by the acidic chemical erosion fill the pores, weakening the crack closure phase and decreasing the cumulative acoustic emission rate values. As erosion continues, calcium leaching leads to an increase in pore structure. Consequently, its deformation rapidly changes from microcrack closure to microcrack expansion, enlarging the macroscopic crack damage and sharply increasing the acoustic emission rate.
3. Chemical erosion ultimately causes the performance of the CGB to deteriorate. However, the stress threshold for microcrack initiation and macro crack extension increases and decreases as the microporous structure changes with the erosion time. After 60 days of immersion, the stress thresholds for microcrack initiation and macrocrack extension increase by 20% and 6%, respectively. However, as the immersion time increases to 150 days, the stress thresholds for microcrack initiation and macrocrack extension decrease by 56% and 16%, respectively.
4. The mechanism of deterioration for CGB due to chemical ion erosion can be divided into two stages. In the first stage, the strength of the CGB is enhanced due to the hydration reaction and ettringite’s filling and compacting effect. Later, the alkaline environment is destroyed, and ettringite accelerates decomposition and dissolution, causing the hydration reaction of the cementitious material to be weaker than the strength deterioration effect caused by acidic corrosion. As the load increases, microcracks are generated at the bond interface, especially the microfractures along the interface transition zone, resulting in strength degradation.

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