

Engineering strength through microstructure: gradation and content effects in cemented high-sulfur tailings-crushed rock backfill

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ABSTRACT

This study addresses the environmental hazards posed by the extensive buildup of crushed rock and sulfur-rich tailings produced during mining operations. A complete lab study was carried out to assess strength performance and internal microstructure of a cemented fill system made from high-sulfur tailings and crushed rock (CHSTCRB). UCS trials and SEM inspections were performed based on an orthogonal experimental design. Key parameters, including UCS, critical damage value (D_{pk}), failure modes, energy dissipation behavior, and microstructural features, were examined. The findings show that UCS declines as the cement-to-tailing (c/t) ratio drops, with factor influence ranked as follows: c/t ratio having the greatest impact, followed by crushed rock gradation, and then content. Proper aggregate design notably boosts strength while easing degradation effects. The deterioration behavior is ruled by a dual effect of cement hydration and pyrite oxidation, exhibiting a distinct three-stage evolutionary process. Prolonged curing increases D_{pk} , progressively weakening the internal microstructure, enhancing porosity, and resulting in a marked reduction in mechanical strength. As rock content increases and particle size distribution becomes optimized, the failure mechanism transitions from a brittle, tensile-dominated fracture to a hybrid tensile-shear mode, characterized by pronounced stratification and extensive, inter-connected crack propagation. Energy evolution analysis shows a continuous rise in total energy, while elastic strain energy follows a rise-fall trend, peaking before falling as structural damage develops. In the interim, dissipated energy escalates sharply in later steps, revealing intensified damage and fracture activity. An elevated c/t share facilitated cemented materials and basic compaction formations. Extended cure periods enhanced ITZ (interfacial transition zone) contact, and rock aggregate well mitigated worsening of high-sulfur tailings. Accordingly, these findings run a solid scientific basis for optimizing design and engineering application of CHSTCRB systems in contemporary mining operations.

1. Introduction

Mineral resources are vigorous strategic assets that have long supported rapid socio-economic development [1]. In recent years, easily accessible deposits have become increasingly depleted. As a result, mining activities have progressively shifted toward deeper resource extraction [2]. Massive amounts of waste rock from mineral extraction occupy significant land areas [3]. Oxidation of sulfide tailings produces acidic wastewater, which acidifies water and soil and leaches heavy metals, causing severe pollution [4–6]. These challenges underscore the pressing need for sustainable management of mining byproducts. One

effective approach to utilizing solid waste is to use unclassified tailings and waste rock as fill aggregates [7,8]. These materials can fill underground mined-out voids, turning waste into a handy resource. This strategy not only reduces surface disposal but also eases environmental impacts [9,10]. Developing effective filling systems has become a pressing technical challenge in the mining industry [11]. Addressing this issue is essential for promoting bearable mining practices and advancing the construction of naturally friendly, “green” mines [12,13].

Over the years, cemented tailings–crushed rock fills have attracted large global research attention, leading to a broad and evolving body of scholarly work. Gao et al. [14] explored how both proportion and size of

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waste rock shape flexural performance and failure patterns of CT-WRB, revealing critical links between material composition and structural behavior. Huang et al. [15] examined how varying WR quantity and grain diameter affect strength, collapse, and microstructure of layered WR-CTB using UCS testing and SEM analysis. Li et al. [16] tried SHPB method to examine CWRB's peak stress, Young's modulus, and energy dissipation characteristics. They further developed a fundamental model to capture the transition behavior between low and high impact strain rate (ISR) regimes. Li et al. [17] performed compressive strength trials on rocks having varying grain diameters and analyzed how these distributions influence WRB's fractal features considering compressive loading. Zhang et al. [18] inspected rock powder type and dosage effects on CTRPB's strength, failure morphology, and energy evolution. They further proposed a damage model to clarify the material's progressive damage evolution.

Similarly, Li et al. [19] performed some strength experiments on CWTB specimens having varying particle size distribution (PSD) and waste rock content (WRC), systematically examined their mechanical response and energy dissipation behavior, and developed a novel damage-based constitutive framework to characterize the corresponding damage evolution law. Li et al. [20] employed a custom bidirectional load order for coarse-sized sources to study how repeated lateral loading affects filled waste rock's compaction, analyzing its impact on lateral/axial strains, void ratio, and the pressure constant. Qiu et al. [21] performed AE trials to assess fill's strength/deformation behavior, using ringing counts, cumulative hits, and r -values to uncover how cement-to-tailing (c/t) proportion and WR quantity influence its strength features. Chen et al. [22] examined how varying waste rock content affects its distribution within cemented backfill. They systematically investigated compressive strength and damage behavior of filling specimens using UCS-AE tests and elucidated the underlying mechanisms of strength development and damage evolution through detailed microstructural analysis.

Substantial attention has also been devoted to high-sulfur tailings fills. He et al. [23] systematically inspected fly ash and sodium bicarbonate combination effect on CHSTF's mechanical performance, microstructural development, and chemical stability. Their study integrated UCS testing, chemical stability evaluation, and microstructural characterization to elucidate the coupled physico-chemical mechanisms governing material behavior. Yin et al. [24] rated strength behavior, flow features, and microstructure of CSTB using fluidity measurements, UCS tests, and SEM-EDS analysis. Wang et al. [25] reviewed how sulfur-rich tailings influence cementitious backfilling's strength, investigated mechanisms behind strength deterioration caused by these tailings, and provided a detailed study of erosive and damaging impacts of sulfides and sulfates. Koc et al. [26] explored how fineness of slags affects strength performance, geochemistry, and long-term stability of high-sulfide tailings CPB over 360 days, using ultrasonic testing and microstructural analysis. Dong et al. [27] used the AAA and NAG procedures to predict static acid potential of tails having four varying sulfur rates and examined specimens' UCS and failure modes over a 28- to 360-day period. Wang et al. [28] performed a comprehensive investigation on high-sulfur CPB, systematically characterizing its rheological and mechanical properties, evaluating multiple performance-control strategies, and assessing associated environmental impacts under different inhibitor regimes. Similarly, Yin et al. [29] conducted UCS experiments on cement-based fill incorporating sulfide-rich tailing, focusing on dual influence of sulfur rate, polypropylene fiber rate, and fiber limit γ on strength development, failure behavior, and damage growth mechanisms. Cheng et al. [30] evaluated effects of varying citric acid content on cemented sulfur tailings fill by comparing its macro-strength and micro-structural features at different curing ages using UCS, XRD, and SEM analyses.

Previous studies have mostly focused on systems containing either crushed rock or high-sulfur tailings in isolation. In contrast, mechanical strength features of cemented high-sulfur tailing-crushed rock backfill

(CHSTCRB) remain largely unexplored, and its underlying strength behavior has yet to be fully elucidated. Accordingly, a systematic investigation of CHSTCRB's strength and microstructural progress is of critical importance for ensuring safe, sustainable and efficient deep mining operations. Collectively, these findings establish a mechanistic framework for the performance-driven design and engineering application of sustainable backfill systems.

2. Materials and methods

2.1. Materials

2.1.1. Rocks

In existing experiments, rock materials were sourced by a copper mine in Xinjiang, retrieved directly from the mine's spoil dump and brought to lab environment for controlled preparation. The rock was fractionated using standard 3-, 6-, and 9-mm sieves to create precise gradation. Fig. 1 illustrates three crushed rock gradations with nominal particle size arrays of 0–3-mm, 3–6-mm, and 6–9-mm. Particles show highly irregular, angular morphologies, characteristics of mechanically fractured natural rock.

Mining site under study is known to contain sulfur-bearing ore. To analyze the mineral composition of fine crushed rock with a grain diameter of 0–3 mm from idem batch, qualitative and semi-quantitative phase analyses were conducted by Empyrean X-ray diffractometer. Prior to testing, samples were rotisserie-dehydrated at 60 °C to eliminate moisture and then pulverized to 200 mesh using a DLS-2000 crusher. XRD analysis was then performed through a 2 θ assortment of 5–90°, employing a scan proportion of 2°/min to depict samples' mineralogy. Besides, chemical composition of 0–3 mm crushed rock was determined using a sequential XRF spectrometer, operated at 60 kV and 140 mA, with a scanning rate of 300°/min.

Fig. 2 illustrates the mineralogical phases and chemical composition of 0–3 mm crushed rock fraction. The results indicate that quartz, pyrite, hematite, andradite, and dickite (2M1) are the principal mineral constituents, with pyrite serving as the dominant carrier of sulfur within the material. XRF analysis revealed that the main oxides were SiO₂ (40.2%), Al₂O₃ (18.7%), SO₃ (11.3%), Fe₂O₃ (10.1%), and CaO (9.4%). SO₃ content of crushed rock was 11.3%, which is equivalent to approximately 4.5% elemental sulfur. This content was at a relatively high level and required attention.

2.1.2. Principal filling ingredients

To ensure experimental consistency, the tailings samples were gained by an underground copper mine in China. Prior to testing, tailing was oven-dried in a 101–0 plug-in thermostatic blast rotisserie at 95 °C for 11-h and subsequently wrapped to prevent wetness reabsorption from the ambient environment. OPC 42.5R, supplied by Shandong Jiuqi Building Materials, was practiced as a cementitious agent, while laboratory tap water was practiced in making specimens. Grain diameters and disseminations of tail and cement were measured via an LSPOP (9) laser sizer, as shown in Fig. 3. Tailing was classified as fine-sized, with a span ratio of 3.00, indicating poor uniformity, but their total gradation was favorable for improving backfill compaction and aggregate density. In contrast, the cement specimen showed a narrower PSD with a span fraction of 1.98, signifying superior consistency. Corresponding D₉₀ and D₅₀ values were 29.7 μ m and 12.7 μ m, respectively, markedly lower than ones of other tail specimens (53.4 μ m and 16.4 μ m). This confirms that binder grains are significantly less and further homogeneously distributed than the tailings.

Fig. 4 presents mineralogy and chemistry of tailing and cement. Tailing is predominantly composed of quartz, pyrite, hematite, and andradite, exhibiting a mineral assemblage like that of the crushed rock. In contrast, binder is mainly making up of alite (C₃S), belite (C₂S), tricalcium aluminate (C₃A), tetracalcium aluminoferrite (C₄AF), and calcite, reflecting its typical clinker-derived phase structure. XRF

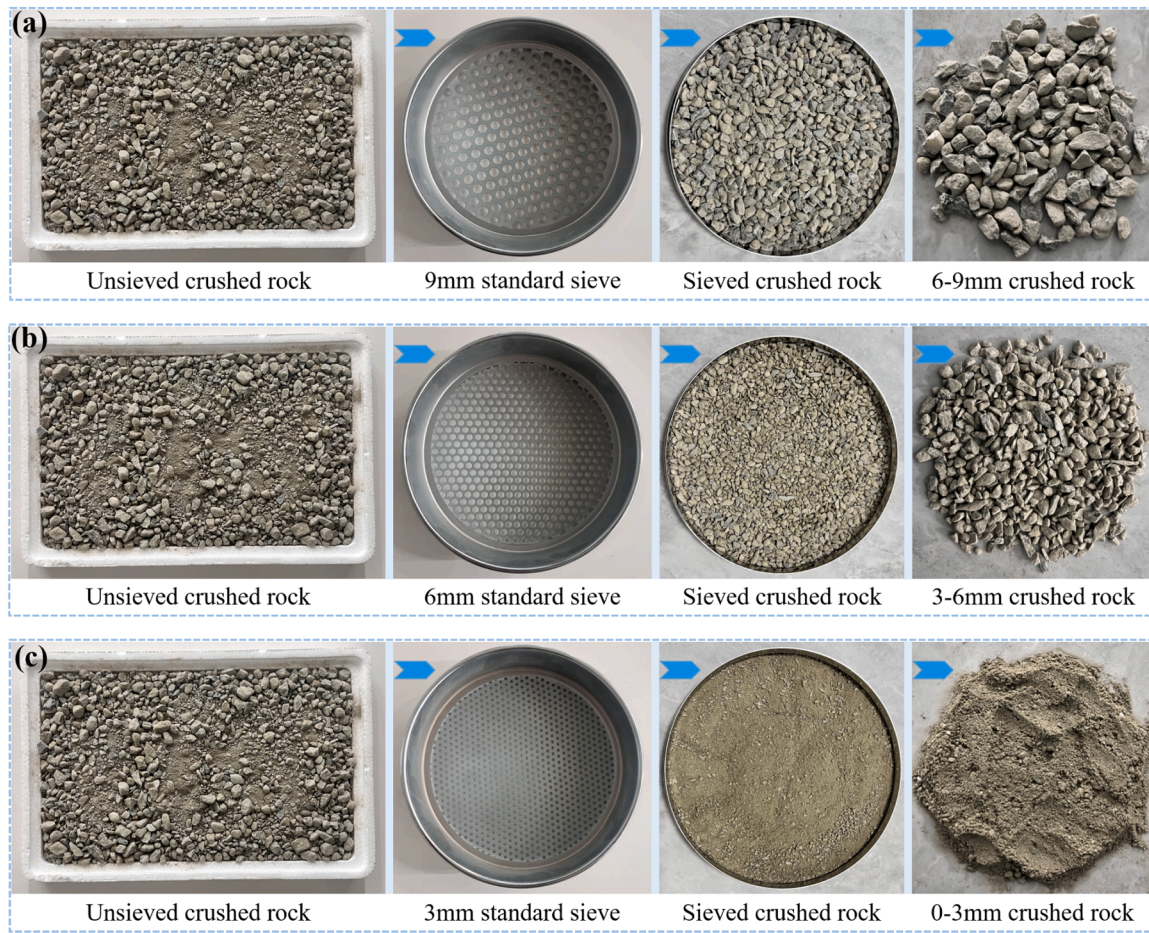


Fig. 1. Crushed rock's sieve analysis procedure and grain size gradation (a: 0–3 mm; b: 3–6 mm; and c: 6–9 mm).

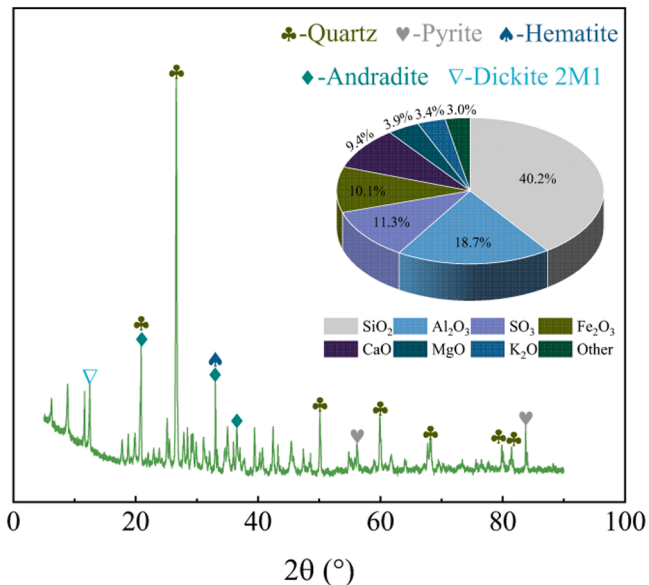


Fig. 2. 0–3 mm crushed rock's mineralogy and chemistry.

analysis showed that the dominant oxides in the tailings were SiO₂ (41.6%), Al₂O₃ (13.4%), SO₃ (20.7%), Fe₂O₃ (13.5%), and CaO (2.8%). Fu et al. [31] found that tailings containing >8% sulfur (i.e., over 20% SO₃) belong to high-sulfur tailings. Thus, tailing employed are

categorized as high-sulfur one due to their elevated pyrite content. The cement's main oxides were SiO₂ (23.9%), Al₂O₃ (10.8%), SO₃ (2.5%), Fe₂O₃ (3.5%), and CaO (55.7%). The cement's high calcium content complements the silicon- and aluminum-rich tailings and crushed rock, enhancing cement hydration and overall backfill performance.

2.2. Orthogonal experiment scheme

To systematically quantify the coupling possessions of c/t proportion, and quantity and gradation of crushed rocks on CHSTCRB's strength features, two sets of three-factor, three-level orthogonal trials were intended based on the L9(3³) orthogonal array (Table 1). This experimental framework enabled the efficient evaluation of individual and interactive influences of the selected variables while minimizing the number of test combinations. The specific factor levels and corresponding mix proportions were defined as follows:

As offered in Table 1, the first orthogonal test series was designed to inspect the effects of three factors on strength behavior of CHSTCRB specimens: c/t ratio (1:4, 1:6, and 1:8), quantity (0%, 10%, and 20%), and gradation (0–3 mm, 3–6 mm, and 6–9 mm) for crushed rocks. To further elucidate the role of coarse aggregate incorporation, a second orthogonal test series was subsequently conducted using the same c/t ratios and gradation ranges, while increasing the crushed rock content to 30%, 40%, and 50%. This experimental design facilitated a systematic evaluation of the individual and combined effects of binder rate, aggregate content, and particle size characteristics on CHSTCRB's mechanical response.

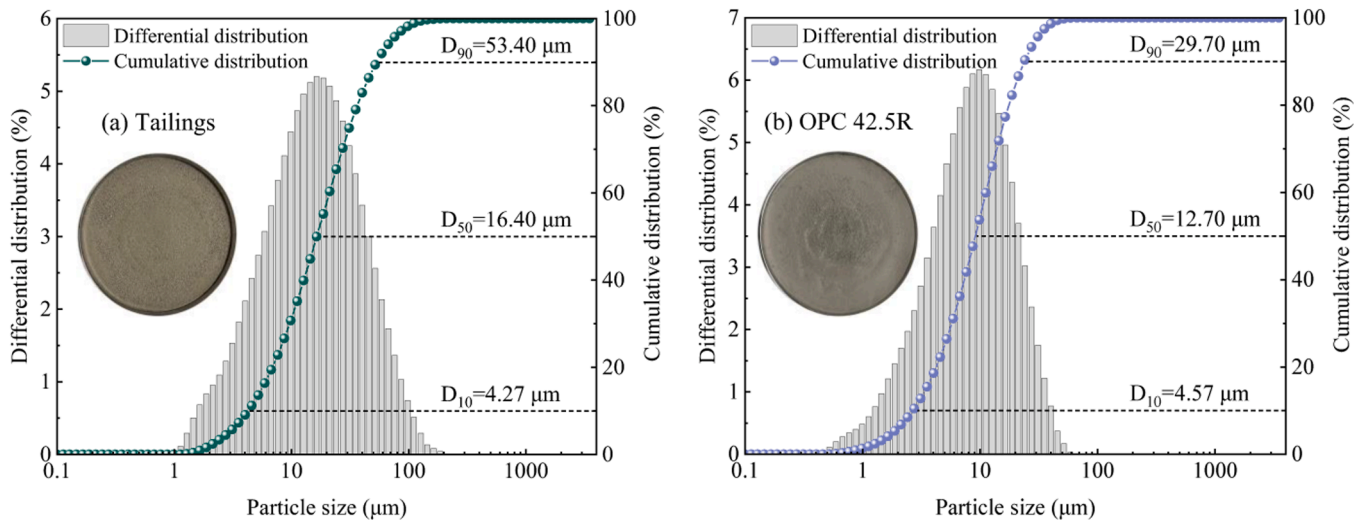


Fig. 3. Grain diameters/spreading of tailing (a) and OPC 42.5R (b).

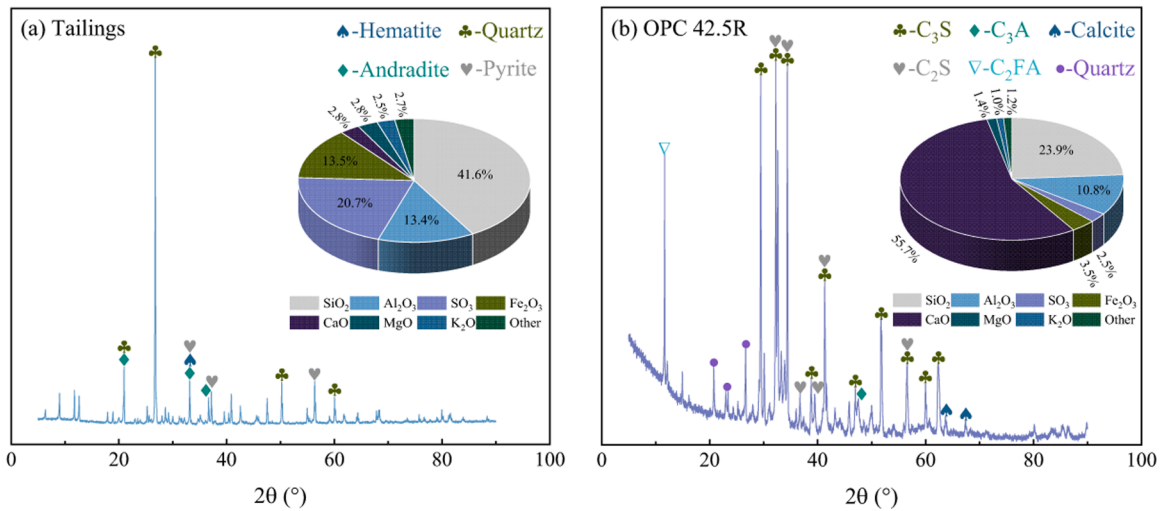


Fig. 4. Mineralogy and chemistry of tailing (a) and OPC 42.5R (b).

Table 1

Mix proportions for the two separate L9(3³) orthogonal arrays designed for low (0–20%) and high (30–50%) rock content ranges.

Group	Level	Affecting parameters		
		c/t proportion	Content of rocks (%)	Gradation of rocks (mm)
Low crushed rock content	1	1:4	0	0–3
	2	1:6	10	3–6
	3	1:8	20	6–9
High crushed rock content	4	1:4	30	0–3
	5	1:6	40	3–6
	6	1:8	50	6–9

2.3. Specimens preparation and experimenting rules

Given the fine particle size of tailings, preliminary tests were conducted prior to formal experiments. The measured slump value was 29.5 cm, and slurry' solid concentration was finally determined to be 68%. c/t ratio of 1:4, 1:6, and 1:8. Water-to-binder ratio (W/B) of 0.425, 0.304 and 0.236. Specimens were cured after 28- and 56-day. All materials were precisely weighed using an automated weightier with 0.01 g

precision. Dry components were initially blended for 3-min, after which water was inserted, and the mix was further homogenized in a JJ-5 mortar blender for an additional 3-min. Unmatured paste was placed in tube-shaped specimen holders pre-coated with release agent and vibrated 1 min until air bubbles ceased emerging. The slurry for specimens in the same group was prepared uniformly and then poured into three separate molds. Fills were dried in an SHBY-40B room at 21 °C and 94% relative moisture, with demolding performed after 48 h. Fig. 5 depicts the entire paste creation workflow.

2.4. UCS experiments

Following relevant cures, CHSTCRBs' ends (diameter × height was 50 × 100 mm) were carefully ground flat to ensure uniform load transfer during uniaxial testing. UCS was measured via a computer-precise electronic testing machine at a dislocation proportion of 1 mm/min [32]. Records were nonstop taped and stored digitally throughout test, while a camera documented specimens' failure progression. To minimize experimental variability, 3 filling materials were tried for every recipe, and median strength was assessed. Fig. 6 demonstrates UCS testing procedure.

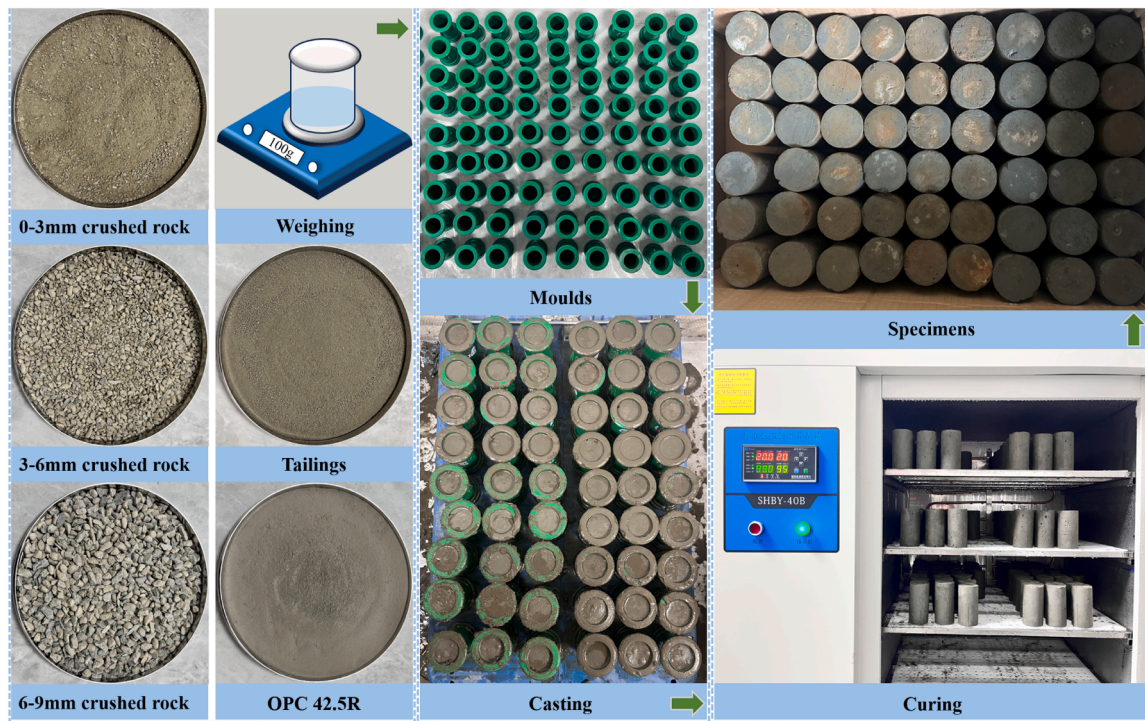


Fig. 5. Paste specimens' creation steps.



Fig. 6. Photos regarding UCS test's application and evaluation.

2.5. Inspection of SEM-EDS analyses

Fig. 7 illustrates the SEM-EDS characterization procedure adopted in



Fig. 7. Creation, curing and testing steps for SEM-EDS specimens.

this study. The microstructure of CHSTCRB was observed via a ZEISS Gemini SEM 300 operating at a hastening voltage of 10–20 kV, an enlargement of 3000 $\times$, and a running space of 8–9 mm. Prior to observation, specimens were dried at 50 $^{\circ}$ C, sputter-coated with platinum to enhance electrical conductivity, and analyzed under vacuum conditions. Integrated EDS and BSE detectors enabled simultaneous acquisition of microstructural features and elemental distribution information, with all data digitally recorded for subsequent analysis.

3. Results and discussion

3.1. Multi-aspects' joint effects on strength performance of specimens

Fig. 8(a) offers UCS of 28-day cured CHSTCRB considering orthogonal test states. Overall, UCS decreased as c/t proportion declined. At c/t proportion of 1:4, fill without crushed rock unveiled a UCS of 2.96 MPa, which augmented to 3.41, 3.31, 3.21, 1.97, and 2.25 MPa with varying crushed rock contents. The relative change rates compared with the group with 0% rock content of 15.2%, 11.8%, 8.4%, –33.4%, and –24.0%, respectively. For c/t proportion of 1:6, UCS results shifted between 1.61 and 2.18 MPa, with relative change rates of 17.4%, –1.7%, 22.5%, 5.6%, and –9.6%. At c/t proportion of 1:8, strengths were generally below 2 MPa, measuring 1.56, 1.65, 1.65, 1.85, 1.84, and 0.74 MPa, with relative change rates of 5.8%, 5.8%, 18.6%, 17.4%, and –52.6%, respectively. These results highlight the combined impact of c/t proportion and crushed rock rate on CHSTCRB's strength development.

Fig. 8(b) presents UCS results of 56-day cured CHSTCRB under orthogonal experimental conditions. Overall, the variation pattern closely resembled that observed at 28 d. For specimens having a c/t proportion of 1:4, UCS varied between 2.59 and 4.49 MPa, exhibiting relative changes of 15.1%, 5.5%, 65.7%, –4.4%, and 26.2% across successive test groups. These results indicate that effect of crushed rock rate and gradation on strength development remained pronounced after prolonged curing. For c/t proportion of 1:6, UCS values varied between 2.17 MPa and 2.66 MPa, with relative change rates of –1.0%, 3.6%, 12.6%, 19.8%, and –2.3%. As c/t proportion was 1:8, strengths were generally lower, shifting between 0.46 and 2.45 MPa, and relative

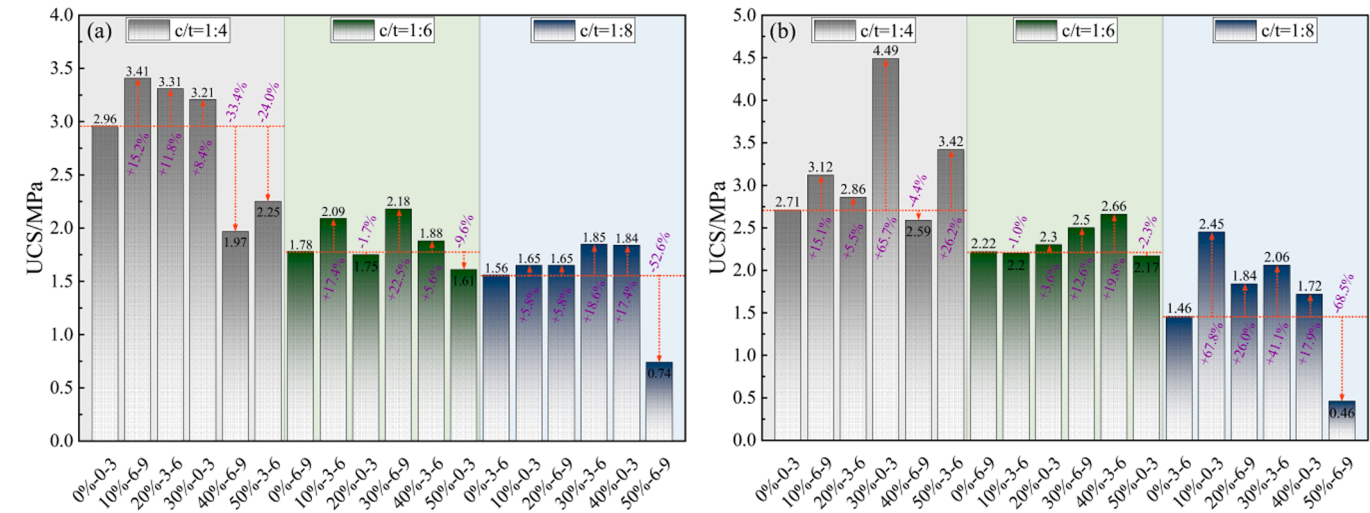


Fig. 8. Uniaxial compressive performance of CHSTCRBs cured at: (a) 28- and b) 56-day.

change rates spanned 67.8%, 26.0%, 41.1%, 17.9%, and –68.5%. These results indicate that, while UCS generally decreases with lower c/t ratios, both age and crushed rock content pointedly affect CHSTCRB’s strength development.

The results reveal a distinct fixed outcome of quantity and gradation of crushed rocks on strengthening of CHSTCRB. In general, UCS increased with increasing crushed rock content and decreasing particle size, suggesting that finer gradations and higher aggregate incorporation enhanced particle packing density, interparticle interlocking, and load-transfer efficiency within the backfill matrix. However, when crushed rock content or gradation was excessively high, particles tended to settle at the bottom, producing a loose structure where cementitious material could not fully occupy the pores, leading to reduced strength. At optimal aggregate ratios, an interlocking framework formed, with hydrated cement filling the voids between particles, substantially improving structural density and UCS. Although the high-sulfur tailings used in this study tended to weaken backfill strength over time, a well-balanced aggregate ratio mitigated this deterioration, demonstrating that the interlocking structure of aggregates can dominate and reduce the material’s sensitivity to strength loss.

The orthogonal test data were analyzed using range analysis to evaluate relative significance of each factor based on response values at different levels. Parameter sensitivity analysis and factor priority ranking were completed, and the relevant results are summarized in Tables 2–5. For 28 d curing in the first group (Table 2), the c/t ratio (1:4, 1:6, 1:8) exhibited the highest influence on UCS with a range value $R = 1.28$, $F = 44.334$, followed by crushed rock gradation (0–3, 6–9, and 3–6

Table 2
Range values and factor significance for UCS.

/	Affecting parameter: 28-day		
	c/t ratio	Content of rocks (%)	Gradation of rocks (mm)
k_1	2.84	2.10	2.12
k_2	1.87	2.16	1.99
k_3	1.56	2.01	2.16
R	1.28	0.15	0.16
Sensitivity	Cement/tailings ratio > Crushed rock gradation > Crushed rock content		
SS	8.002	0.109	0.131
df	2	2	2
MS	4.001	0.054	0.065
F	44.334	0.602	0.724
P	<0.001	0.558	0.498
Contribution rate	80.37%	1.09%	1.31%
Error	SS = 1.715, df = 19, MS = 0.090, Contribution rate = 17.22%		

Table 3
Range values and factor significance for flexural UCS.

/	Affecting parameter: 28-day		
	c/t ratio	Crushed rock content (%)	Crushed rock gradation (mm)
k_1	2.48	2.41	2.32
k_2	1.99	1.90	1.63
k_3	1.48	1.63	1.99
R	1.00	0.78	0.68
Sensitivity	Cement/tailings ratio > Crushed rock content > Crushed rock gradation		
SS	4.500	2.857	2.103
df	2	2	2
MS	2.250	1.428	1.052
F	34.284	21.762	16.020
P	<0.001	<0.001	<0.001
Contribution rate	41.78%	26.52%	19.52%
Error	SS = 1.313, df = 20, MS = 0.066, Contribution rate = 12.19%		

Table 4
Range values and factor significance for UCS.

/	Affecting parameter: 56-day		
	c/t ratio	Crushed rock content (%)	Crushed rock gradation (mm)
k_1	3.01	2.24	2.59
k_2	2.24	2.59	2.39
k_3	1.92	2.33	2.17
R	1.09	0.35	0.42
Sensitivity	Cement/tailings ratio > Crushed rock gradation > Crushed rock content		
SS	5.651	0.598	0.799
df	2	2	2
MS	2.825	0.299	0.399
F	25.106	2.655	3.548
P	<0.001	0.096	0.049
Contribution rate	61.52%	6.51%	8.70%
Error	SS = 2.138, df = 19, MS = 0.113, Contribution rate = 23.28%		

mm) with $R = 0.16$, $F = 0.724$ and rock content (0%, 10%, 20%) with $R = 0.15$, $F = 0.602$. In the second group at 28 d (Table 3), the c/t ratio remained the most significant factor ($R = 1.00$, $F = 34.284$), followed by crushed rock content ($R = 0.78$, $F = 21.762$) and gradation ($R = 0.68$, $F = 16.020$). For specimens cured 56 d in the first group (Table 4), the c/t

Table 5
Range values and factor significance for UCS.

/	Affecting parameter: 56-day		
	c/t ratio	Content of rocks (%)	Gradation of rocks (mm)
k_1	3.37	2.79	2.66
k_2	2.44	2.32	1.85
k_3	1.32	2.02	2.62
R	2.05	0.77	0.82
Sensitivity	Cement/tailings ratio > Crushed rock gradation > Crushed rock content		
SS	18.992	2.733	3.785
df	2	2	2
MS	9.496	1.366	1.893
F	61.071	8.788	12.172
P	<0.001	0.002	<0.001
Contribution rate	67.09%	9.65%	13.37%
Error	SS = 2.799, df = 18, MS = 0.155, Contribution rate = 9.89%		

ratio again dominated ($R = 1.09$, $F = 25.106$), with crushed gradation ($R = 0.42$, $F = 3.548$) and rock content ($R = 0.35$, $F = 2.655$) showed progressively lower influence. In the second group at 56 d (Table 5), the c/t ratio had a range of $R = 2.05$, $F = 61.071$, followed by crushed rock gradation ($R = 0.82$, $F = 12.172$) and content ($R = 0.77$, $F = 8.788$). These results highlight the consistent dominance of c/t ratio in controlling UCS, with crushed rock content and gradation playing secondary roles.

The range analysis results indicate that, despite 28- and 56-day curing age, influence of investigated factors on CHSTCRB's strength consistently tracked as follows: c/t proportion > rock gradation > rock

content, with the c/t ratio serving as the primary controlling parameter. Specifically, increasing the c/t ratio from 1:8 to 1:4 resulted in average UCS increases from 1.62 to 3.23 MPa, 1.48 to 2.48 MPa, 1.92 to 2.90 MPa, and 1.41 to 3.50 MPa, corresponding to strength enhancements of 99.4%, 67.6%, 51.0%, and 148.2%, respectively. In contrast, the effect of crushed rock content was comparatively moderate but became increasingly pronounced at higher incorporation levels. For specimens containing 0–20% crushed rock, average strength variations were limited to 11.8% and 17.8% at 28 d and 56 d, respectively, whereas increasing the rock content to 30–50% amplified the corresponding variations to 36.5% and 33.1%. Furthermore, a clear interaction between crushed rock content and gradation was observed. At low rock contents, changing the gradation from 0–3 mm to 6–9 mm produced relatively small strength fluctuations of 8.6% and 12.9%, while at higher rock contents the fluctuations increased markedly to 26.6% and 33.7%. These results validate that impacts of quantity/gradation of crushed rocks on CHSTCRB's strengthening are strongly coupled and nonlinear, with their influence becoming increasingly significant as aggregate incorporation increases. The combined action of these factors governs particle packing characteristics, load-transfer efficiency, and ultimately the macroscopic mechanical performance of the backfill.

Fig. 9 illustrates the impacts of diverse factor levels on strength progress of CHSTCRB. As shown in Fig. 9(a), for specimens containing 0–20% crushed rock and cured for 28 d, optimal parameter combination was a c/t proportion of 1:4, a rock quantity of 10%, and a rock aggregate of 3–6-mm. For fills having higher contents (30–50%) of crushed rocks at idem cure period (Fig. 9(b)), optimal combination shifted to a c/t ratio of 1:4, a crushed rock content of 30%, and a finer gradation of 0–3 mm.

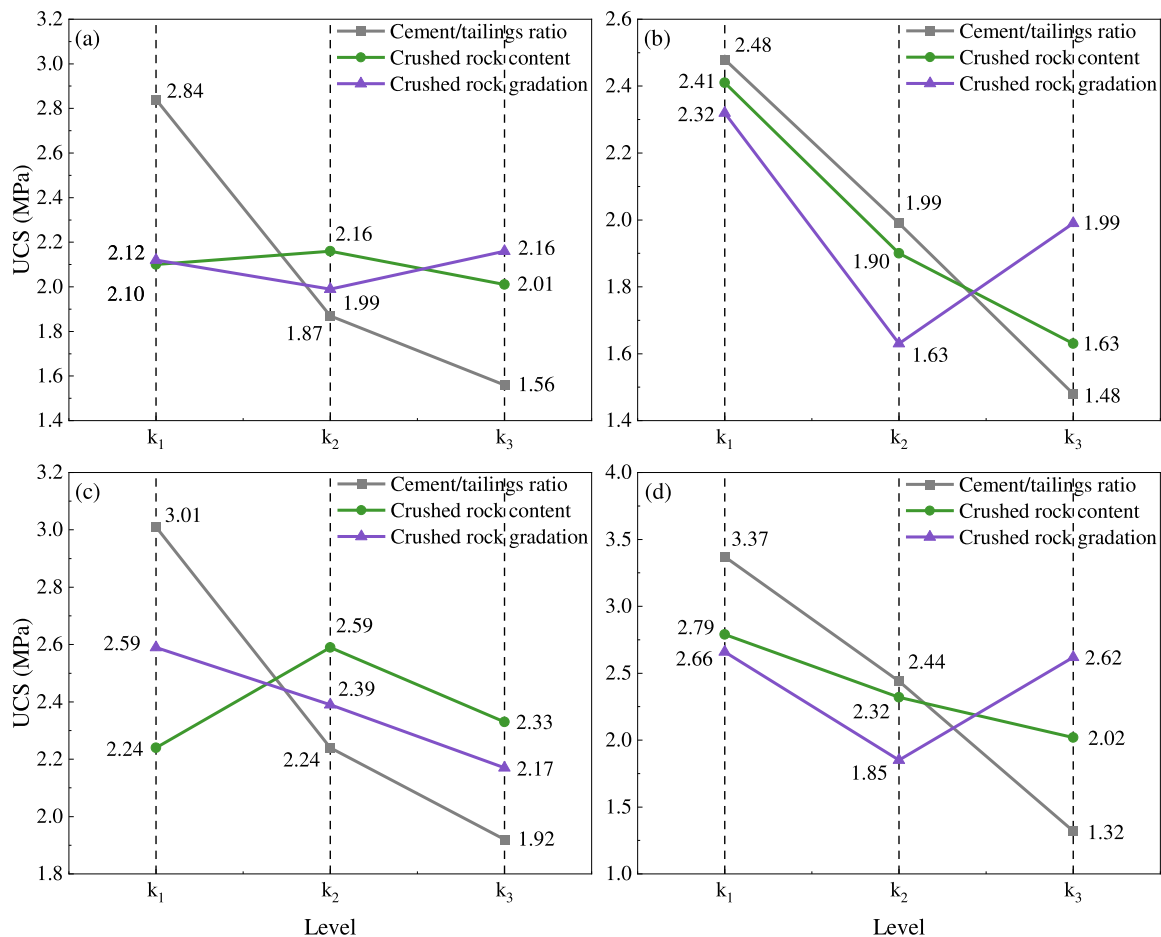


Fig. 9. Variation of UCS with factor levels under diverse cure periods and crushed rock rates: (a) 28 d, 0–20% crushed rock; (b) 28 d, 30–50% crushed rock; (c) 56 d, 0–20% crushed rock; (d) 56 d, 30–50% crushed rock.

After 56 d of curing, the optimal c/t ratio and crushed rock content remained unchanged for both content ranges, whereas the optimal gradation for low-content specimens changed from 3–6 mm to 0–3 mm (Fig. 9(c)). Similarly, for high-content specimens, optimal combination consisted of a c/t part of 1:4, an amount of 30%, and a 0–3-mm gradation (Fig. 9(d)) for crushed rocks. These results ultimately indicate that inputs of crushed rock content/gradation to UCS are strongly coupled and nonlinear. An apt aggregate amount and PSD enhance particle packing and interlocking, improve the effectiveness of hydration products, and consequently promote CHSTCRB's strength development.

3.2. Damage factor associated with high-sulfur deterioration

Building on earlier analyses of sulfur content in tailings and crushed rock and considering rock content and gradation effects on backfill deterioration, this section examines the strength degradation of high sulfur backfill without crushed rock. When sulfide minerals such as pyrite are present, two primary reactions occur simultaneously within the backfill: cement hydration and pyrite oxidation. After slurry preparation and setting (Fig. 10(a)), these reactions interact and constrain one another, producing age-dependent variations in mechanical behavior (Fig. 10(b)).

The coupled hydration-oxidation mechanism underlying high-sulfur fill degradation can be divided into three successive stages: *Hydration-dominated stage*: Initially, cement hydration proceeds much faster than pyrite oxidation. Hydration products such as calcium silicate gradually fill large pores, enhancing the overall compactness and leading to continuous increases in compressive strength. *Transition stage*: As hydration slows, the production of cementitious compounds becomes limited, the fraction of harmless pores declines, and harmful pores gradually increase. This results in minor fluctuations in backfill strength, which may slightly rise or fall. *Oxidation-dominated stage*: Eventually, pyrite oxidation surpasses cement hydration. Sulfate and hydrogen ions react with hydrated cementitious products, causing decomposition and the formation of expansive substances. Internal stress rises, harmful and multi-harmful pores increase sharply, and fill's strength decreases notably [33]. The increase in macro-pore diameter observed during the curing process provides structural evidence of internal damage. While this microstructural degradation aligns well with the characteristics of a deterioration process dominated by pyrite oxidation, it cannot be definitively isolated from other physical factors such as autogenous shrinkage or drying shrinkage. Therefore, while the current three-stage model provides a reasonable phenomenological framework, direct chemical evidence (specifically the temporal evolution of pore water pH and SO_4^{2-} concentrations) is still required in future studies for absolute

corroboration.

To capture the progressive degradation of CHSTCRB arising from sulfur-related deterioration, its stress–strain response and damaging progress under compression were explored. The extent of damage was characterized by damaging flexible D , which is clear as follows [33]:

$$\sigma = E\varepsilon(1 - D) \quad (1)$$

where, E denotes the slope of the initial linear elastic segment of the stress–strain curve, σ is stress, ε is strain, and D is damaging factor. By definition, a higher D value corresponds to a greater proportion of internal defects within the backfill under compressive loading, reflecting more advanced material degradation. It was worth noting that the initial linear elastic modulus obtained from monotonic tests was a widely accepted empirical approximation for damage modeling of backfill materials [34]. This method assumed an initial macroscopic damage state of zero and focused on investigating the relative evolution of damage under external loads.

When $\sigma = \sigma_{pk}$, D_{pk} represents the critical damage value. Fig. 11 presents fill's critical damage values during strength deterioration stage at 28- and 56-day, as derived from experimental calculations. As shown in Fig. 11(a), for a c/t share of 1:4, 28- and 56-day cured specimens' D_{pk} values were 0.387 and 0.542, respectively, corresponding to an increase of 40.1%. At a c/t proportion of 1:6, D_{pk} rose from 0.388 to 0.693, yielding a progress degree of 78.6%. For a c/t ratio of 1:8, values were 0.398 and 0.420, indicating a relatively small increase of 5.5%. Fig. 11 (b) further illustrates c/t proportion effect on D_{pk} . At 28 d curing, D_{pk} increased slightly from 0.387 to 0.398 as c/t proportion changed from 1:4 to 1:8, having marginal growth rates of 0.3% and 2.6%. At 56 d, D_{pk} values of 0.542, 0.693, and 0.420 were obtained for c/t rates of 1:4, 1:6, and 1:8, respectively, corresponding to changes of 27.9% and –22.5%. These results highlight a complex, non-monotonic interaction between cement content, age, and damage evolution behavior.

The results demonstrated that the peak damage coefficient for the 1:6 specimen cured for 56 d reached 0.693. Unlike brittle rocks, CTB is highly porous and exhibits prominent pseudo-ductility. Prior to reaching macroscopic peak stress, the material undergoes extensive pore compaction, micro-crack coalescence, and plastic yielding, which leads to a substantial increase in pre-peak strain. Similar high peak damage values (ranging from 0.5 to 0.8) have been widely reported in CTB damage evaluation studies [34]. Meanwhile, curing time had a significant influence on D_{pk} , with values increasing progressively as hydration proceeded. In strength deterioration's later stage, sulfide-induced corrosion weakened internal structure of 56 d-cured high-sulfur tailings fill compared with 28 d specimens. As a result, even under relatively low stress, pore coalescence and microcrack propagation were accelerated, leading to an increase in the critical damage threshold and a

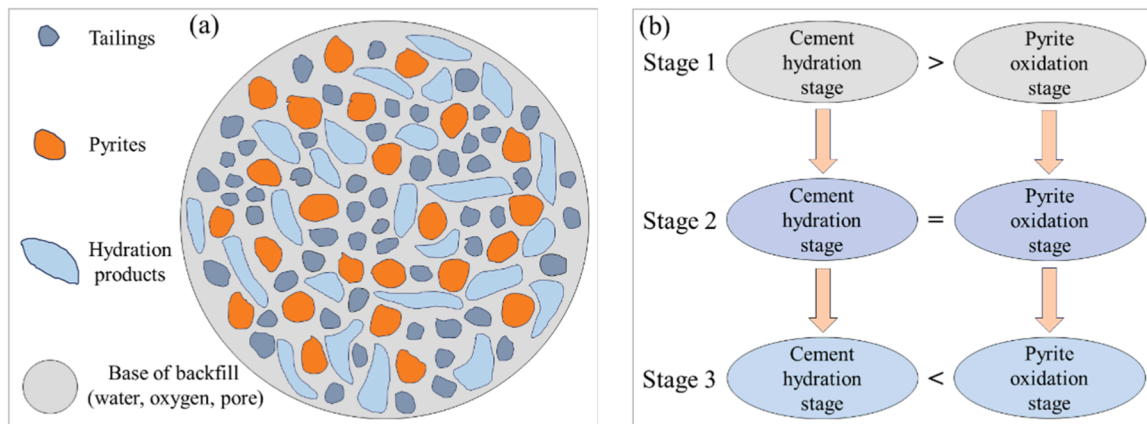


Fig. 10. Schematic illustration of the deterioration and damage mechanisms of high-sulfur backfill: (a) internal microstructure; (b) major reaction pathways involved in degradation.

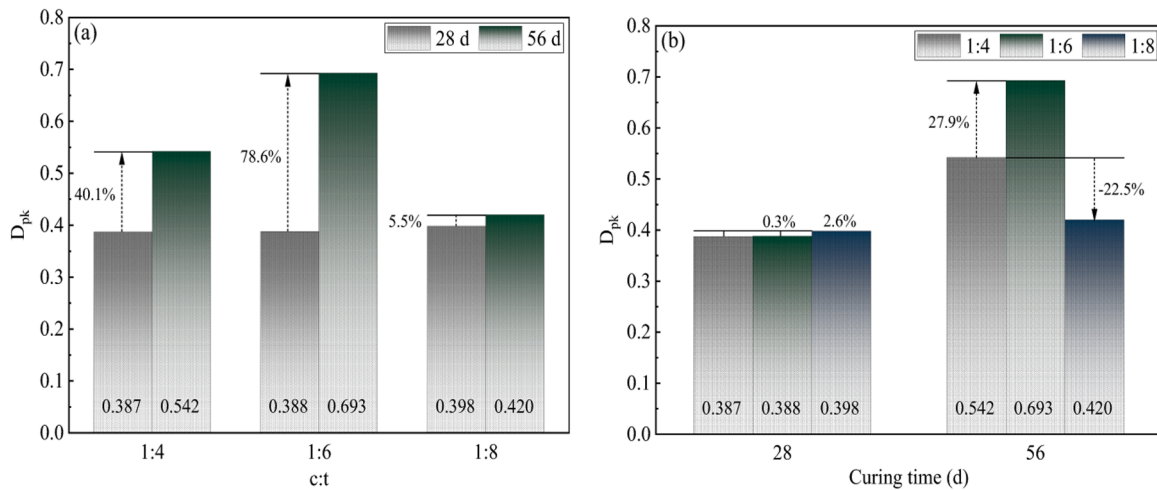


Fig. 11. Variation in D_{pk} as a function of age and c/t proportion: (a) curing time; (b) c/t ratio.

corresponding reduction in overall mechanical integrity [33]. SEM observations further corroborated this behavior, revealing a higher density of original pores and cracks and larger defects in specimens at the later stages of deterioration.

3.3. Analysis of failure modes

To quantitatively characterize the macroscopic failure modes, the fracture angle was determined using digital image processing. High-resolution photographs of the failed specimens were analyzed using ImageJ software. The final standard measurement error was approximately $\pm 1^\circ$. Figs. 12 and 13 present failure modes of 28- and 56-day cured CHSTCRBs, respectively. Among investigated factors in orthogonal design, crushed rock content/gradation employed the weightiest influence on ultimate fracture morphology. As shown in Fig. 12(a)-(c), for a c/t proportion of 1:4, measured principal fracture angles (θ) were 74° , 82° , 82° , 84° , 83° , and 88° , respectively. At a c/t proportion of 1:6, corresponding θ values were 82° , 74° , 82° , 84° , 84° , and 87° . For a c/t ratio of 1:8, fracture angles ranged from 74° to 90° , specifically 74° , 90° , 89° , 87° , 86° , and 89° . These results indicate that both crushed rock content and particle size distribution strongly govern fracture geometry under compressive loading, leading to noticeable variations in crack inclination and failure mode.

Fig. 13(a)-(c) exhibit failing styles of CHSTCRBs cured for 56 d. For c/t proportion of 1:4, principal fracture angles (θ) ranged from 83° to 89° , specifically 86° , 83° , 89° , 86° , and 89° . At c/t proportion of 1:6, θ values varied between 69° and 86° (84° , 82° , 83° , 86° , 69° , and 88°). For c/t proportion of 1:8, the angles showed wider variation, ranging from 50° to 88° (88° , 50° , 76° , 79° , 68° , and 88°). Notably, specimens “40%–3–6 mm,” “10%–0–3 mm,” and “40%–0–3 mm” exhibited small main fracture angles of 69° , 50° , and 68° . This was principally triggered by deterioration of ITZ between aggregate and matrix. Sulfate ions released from pyrite oxidation accumulated in the ITZ and formed expansive secondary minerals such as ettringite. Local expansion induced microcracks and greatly reduced the interfacial cohesion [35]. Accordingly, macroscopic failure propagated preferentially along these weakened ITZ boundaries, which accelerated shear failure.

The results revealed that CHSTCRB specimens with low rock content and coarse size predominantly failed in a brittle tensile mode, with axial cracks propagating through the entire specimen. As rock content and size augmented—for example, in fills “40%–6–9 mm” and “50%–3–6 mm”—failing style transitioned to a tensile–shear fused pattern. In these cases, axial tensile cracks appeared in the upper portion, while oblique shear cracks developed in the lower region. This behavior was

influenced by the sedimentation of crushed rock particles, which induced axial stratification: the upper layers were rich in tailings and fine particles, whereas the lower layers consisted of accumulated coarse particles. Upon complete failure, the specimens exhibited noticeable expansion and fragmentation, reflecting the complex interplay between particle distribution and mechanical response.

3.4. Energy dissipation features of filling specimens

Backfill specimens underwent deformation under uniaxial compression, as illustrated in Fig. 14. Assuming an adiabatic condition with negligible heat exchange between fill and external environment, total energy input from external work is denoted as U . Based on the first law of thermodynamics, energy balance is given below [36]:

$$U = \int_0^\epsilon \sigma d\epsilon \quad (2)$$

$$U_e = \frac{1}{2E_0}\sigma^2 \quad (3)$$

$$U_d = U - U_e \quad (4)$$

Here, U denotes total input energy, U_d represents dissipated energy, and U_e is elastic strain energy. E_0 refers to early Young's modulus, while ϵ and σ denote strain and stress, respectively.

In this section, CHSTCRB specimens cured for 28 and 56 days with c/t proportions of 1:4, 1:6, and 1:8 were nominated for energy evolution analysis. Representative samples, including those without crushed rock, as well as those exhibiting the highest and lowest UCS values, were selected for detailed evaluation. The energy–strain relationships are presented in Figs. 15 and 16.

The total input energy U increased monotonically as the external load was applied, while the elastic strain energy U_e followed the stress–strain curve, exhibiting an “increase first, then decrease” pattern. The dissipated energy U_d evolved more slowly at the early stage but rose sharply in the later stages. Based on stress–strain and energy growth features, damage process of CHSTCRB was categorized as 4 distinct steps: pore compaction, elastic distortion, plastic development, and post-peak collapse.

To further explore the energy evolution characteristics, quantitative analyses were conducted on the peak elastic strain energy (U_e^p) and peak dissipated energy (U_d^p) of 28- and 56-day specimens. For fillings containing a c/t part of 1:4 and “0%–0–3 mm” at the curing age of 28 d, the U_e^p reached $10.16 \text{ kJ}\cdot\text{m}^{-3}$ while the U_d^p was only $2.74 \text{ kJ}\cdot\text{m}^{-3}$, with U_e^p/U_d^p of 3.71. This result indicated that high c/t generated sufficient hydration

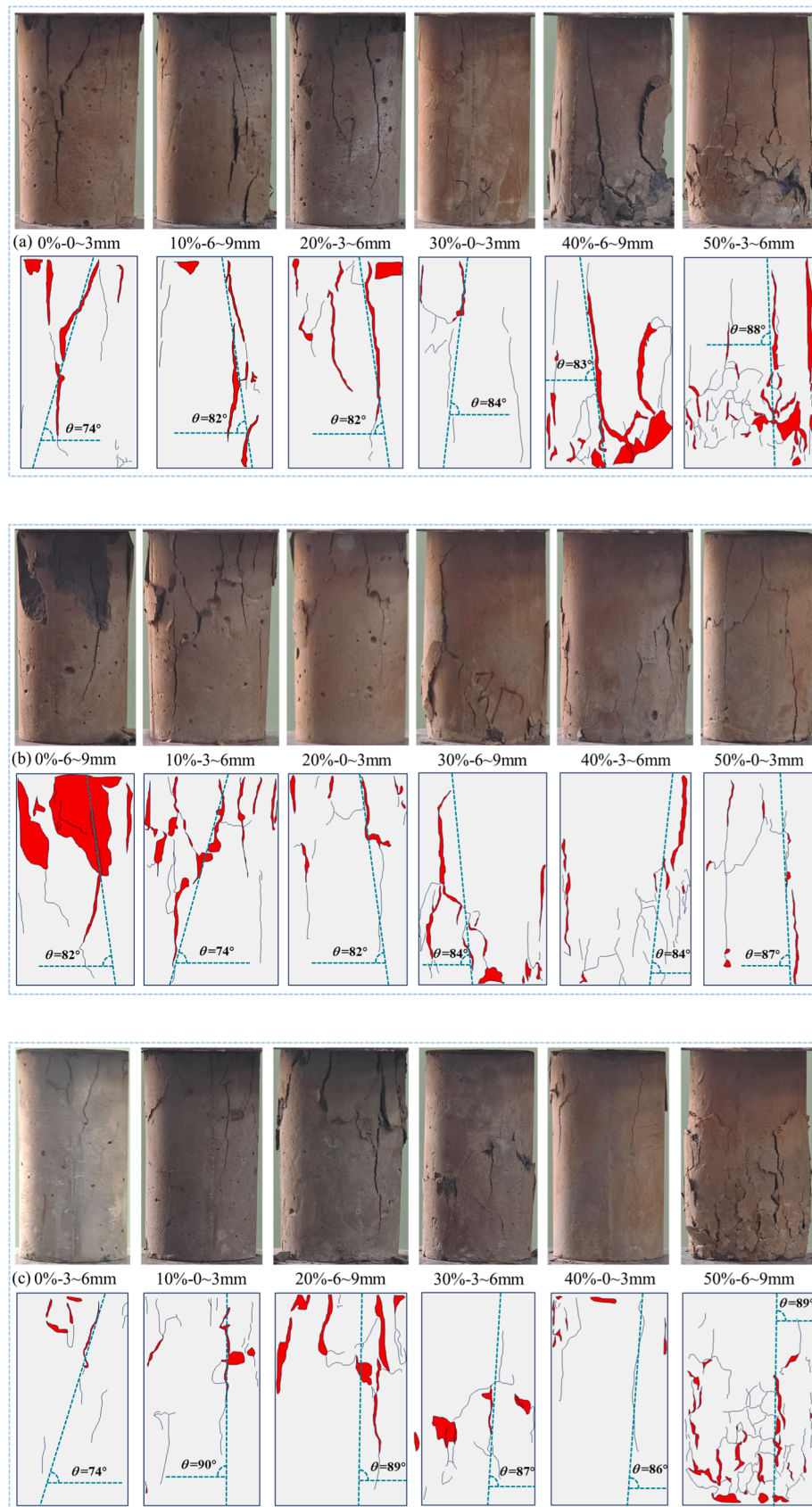


Fig. 12. Failure modes of 28-day cured specimens having a c/t ratio of (a) 1:4; (b) 1:6; and (c) 1:8.



Fig. 13. Failure modes of 56-day cured specimens having a c/t ratio of (a) 1:4; (b) 1:6; and (c) 1:8.

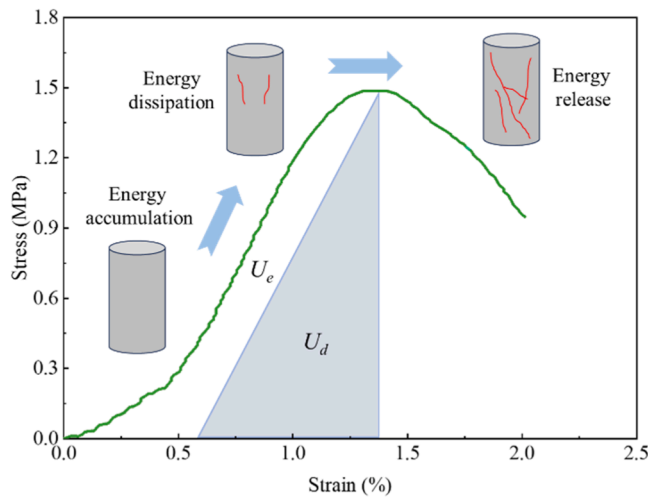


Fig. 14. Schematic diagram of energy calculation for backfill.

products to fully encapsulate aggregates, enabling the backfill to store a large amount of elastic energy before brittle failure occurred. In contrast, when the c/t decreased to 1:8, the specimen with “50%–6–9 mm” exhibited an extremely low U_e^p of $0.61 \text{ kJ}\cdot\text{m}^{-3}$ and an extremely

high U_d^p of $13.08 \text{ kJ}\cdot\text{m}^{-3}$, corresponding to U_e^p/U_d^p of 0.05. This phenomenon reflected a high-energy-dissipation state caused by inherently weak bonding interfaces. After 56-day curing, the U_d^p of all mix proportions increased. Taking fillings covering c/t proportion of 1:4 as a case, their U_d^p rose from $2.74 \text{ kJ}\cdot\text{m}^{-3}$ to $14.72 \text{ kJ}\cdot\text{m}^{-3}$. Distinct energy variation across curing ages verified that expansive secondary products made by continuous oxidation of pyrite severely degraded matrix stiffness and interfacial cohesion. Most of the external mechanical work was dissipated through frictional sliding and frictional resistance along newly formed microdefects, rather than being stored as recoverable elastic strain energy. This energy dissipation mechanism also accounts for the observed change in macroscopic failing style from tensile splitting to shear-dominated one [37].

3.5. Microstructural characteristics analysis

Figs. 17 and 18 present SEM observations of CHSTCRB with no crushed rock at 28- and 56-day, respectively. Systematic assessment of 28-day images revealed a clear trend of microstructural degradation as the c/t proportion diminished from 1:4 to 1:8. The overall Ca/Si ratio decreased from 2.05 to 1.60, while sulfur content increased from 6.10% to 9.39%. Concurrently, the content of hydration products beneficial to strength development—including acicular ettringite (Aft), flocculent C-S-H gel, and clustered AFm phases—decreased markedly. In contrast, unbonded pores and plate-like $\text{Ca}(\text{OH})_2$ crystals became more prevalent,

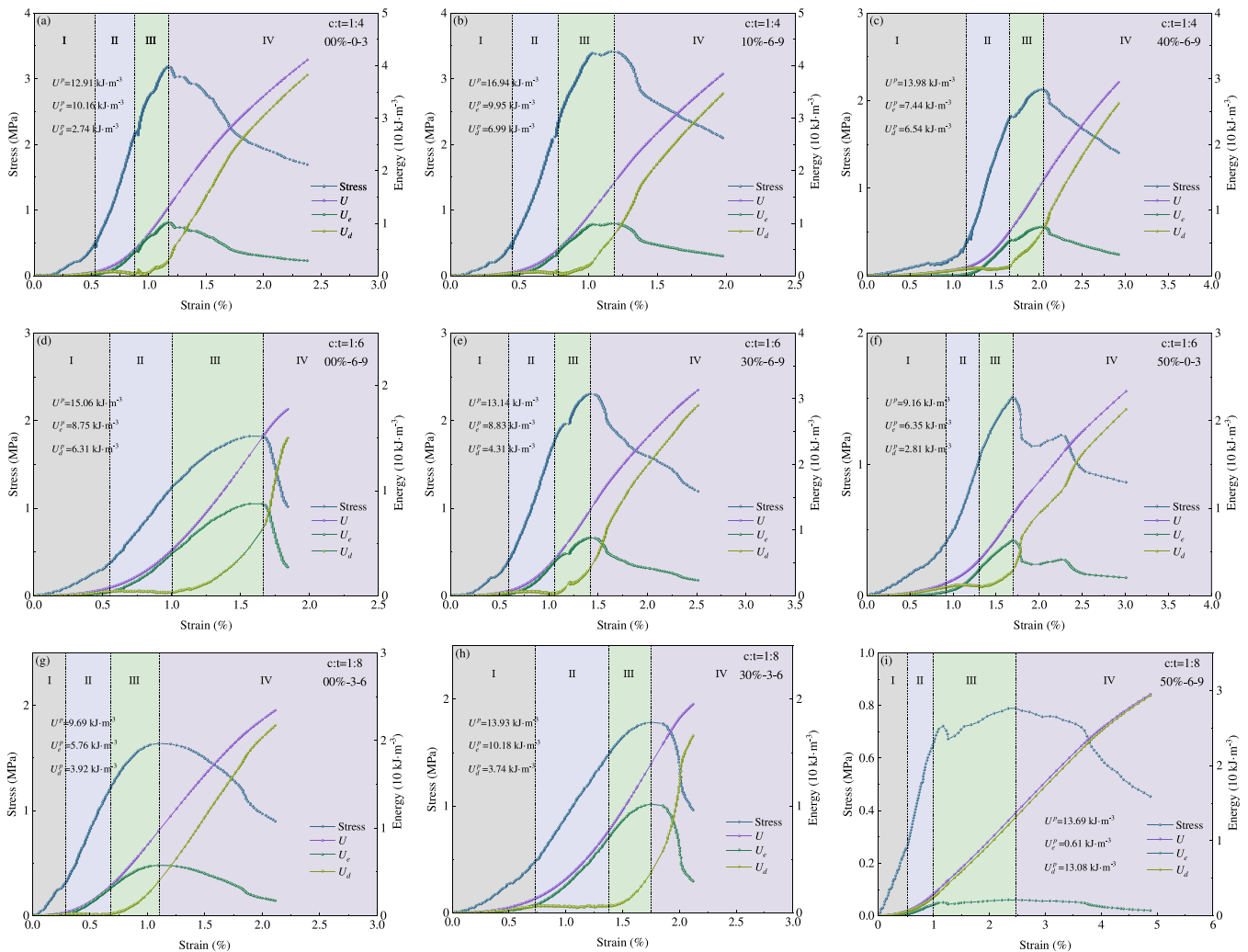


Fig. 15. Variation of energy with strain at 28-day curing age.

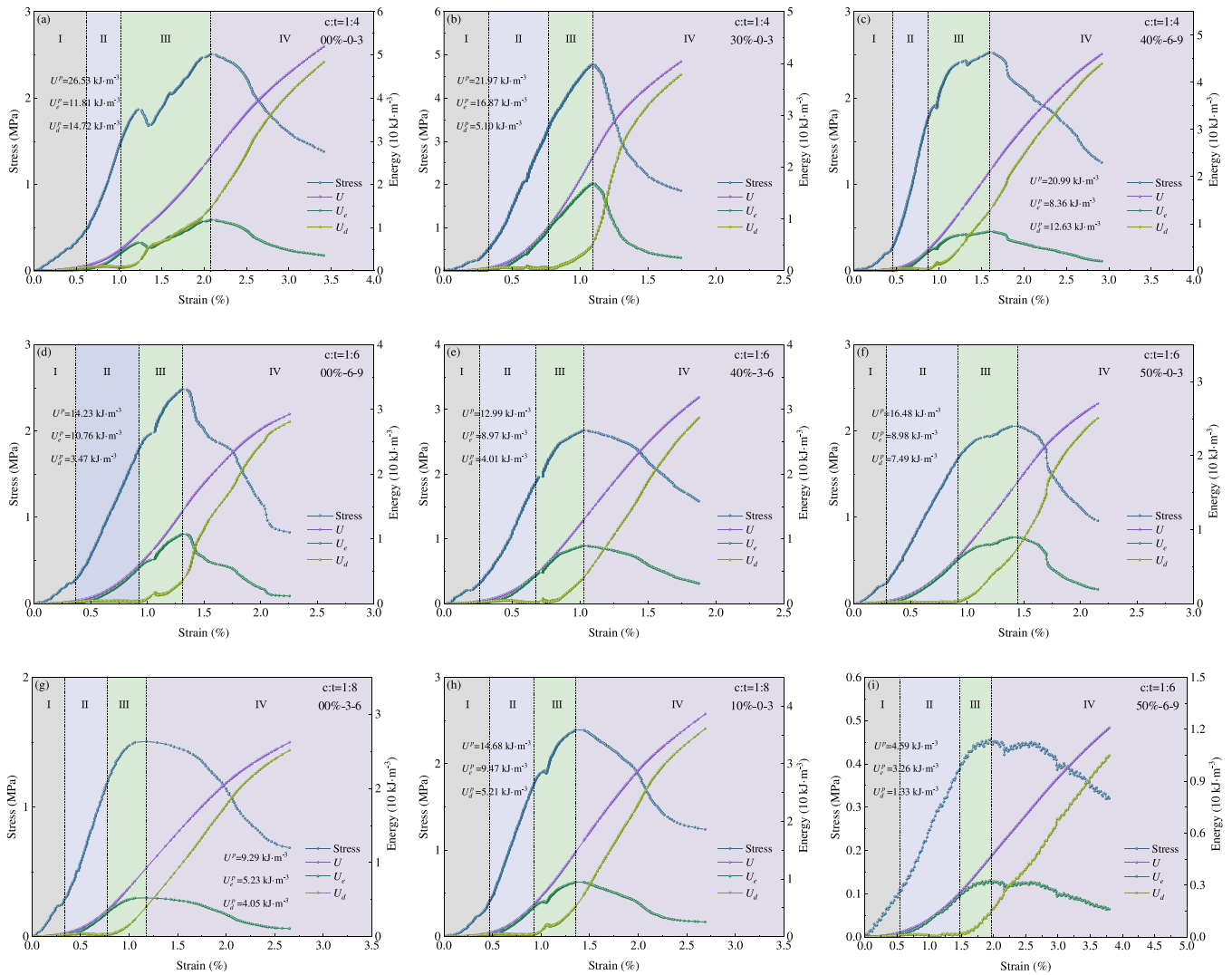


Fig. 16. Variation of energy with strain at 56-day curing age.

indicating a pronounced negative correlation with the macroscopic mechanical properties.

At an extreme c/t proportion of 1:4, densely interwoven acicular-rod Aft crystals formed a continuous network that bridged internal gaps between tailing particles, effectively filling microdefects. Concurrently, flocculent C-S-H gel enveloped the dispersed tailings, creating a stable, compact cementitious system that provided robust support. As the curing period increased to 56 days, the measured Ca/Si ratios for the 1:4, 1:6, and 1:8 mixtures were 3.35, 2.56, and 2.22, respectively, with corresponding sulfur contents of 11.06%, 7.94%, and 14.90%. As the curing age reached 56 d, SO_4^{2-} generated by the oxidation of sulfur-bearing minerals reacted with calcium ions within the system, precipitating abundantly around microcracks, pores, or C-S-H gels to form secondary phases, such as $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ or Aft. This extensive precipitation of Ca- and S-rich secondary products effectively explained the elevated Ca/Si ratios and the significant increase in overall sulfur content observed in the EDS scanning areas. Concurrently, in specific micro-regions where localized depletion of sulfates occurred, some unstable Aft crystals altered into thermodynamically stable AFm crystals, optimizing spatial distribution of the hydration products. The continued generation of C-S-H further reinforced particle encapsulation, synergistically enhancing the internal density.

In contrast, at a low c/t proportion of 1:8, the severe binder deficiency led to the excessive precipitation of platy $\text{Ca}(\text{OH})_2$ crystals

arranged in irregular layouts. Unlike C-S-H gel, these platy crystals lacked sufficient binding capacity, resulting in a loose internal structure dominated by interconnected pores. Comparing the 1:8 specimens over time, the continued accumulation of platy $\text{Ca}(\text{OH})_2$ failed to effectively fill the capillary voids. Instead, the proliferation of these structurally weak crystals, combined with the lack of cohesive C-S-H gel, allowed the original pores to persist and secondary microcracks to propagate along weak interfaces. These observations confirm that, in the later stages of fill deterioration, the coalescence of inherent pores and newly formed microcracks plays a critical role in microstructural degradation, providing a key micromechanical explanation for the long-term reduction in mechanical performance.

Fig. 19 illustrates 28-day cured CHSTCRBs' micromorphological characteristics, with emphasis on ITZ contact between crushed rock particles and cemented medium. Representative specimens exhibiting the highest and lowest UCS values under c/t proportions of 1:4, 1:6, and 1:8 were chosen to investigate ITZ features and their relationship with macroscopic strength.

The measured ITZ sizes of specimens "1:4-10%-6-9 mm," "1:4-40%-6-9 mm," "1:6-30%-6-9 mm," "1:6-50%-0-3 mm," and "1:8-30%-3-6 mm" were 26.8-14.0 μm , 15.1-5.7 μm , 32.7-14.6 μm , 47.8-32.7 μm , and 30.4-16.0 μm , respectively, agreeing to strength values of 3.41 MPa, 1.97 MPa, 2.18 MPa, 1.61 MPa, and 1.85 MPa. These results demonstrate a strong correlation between ITZ

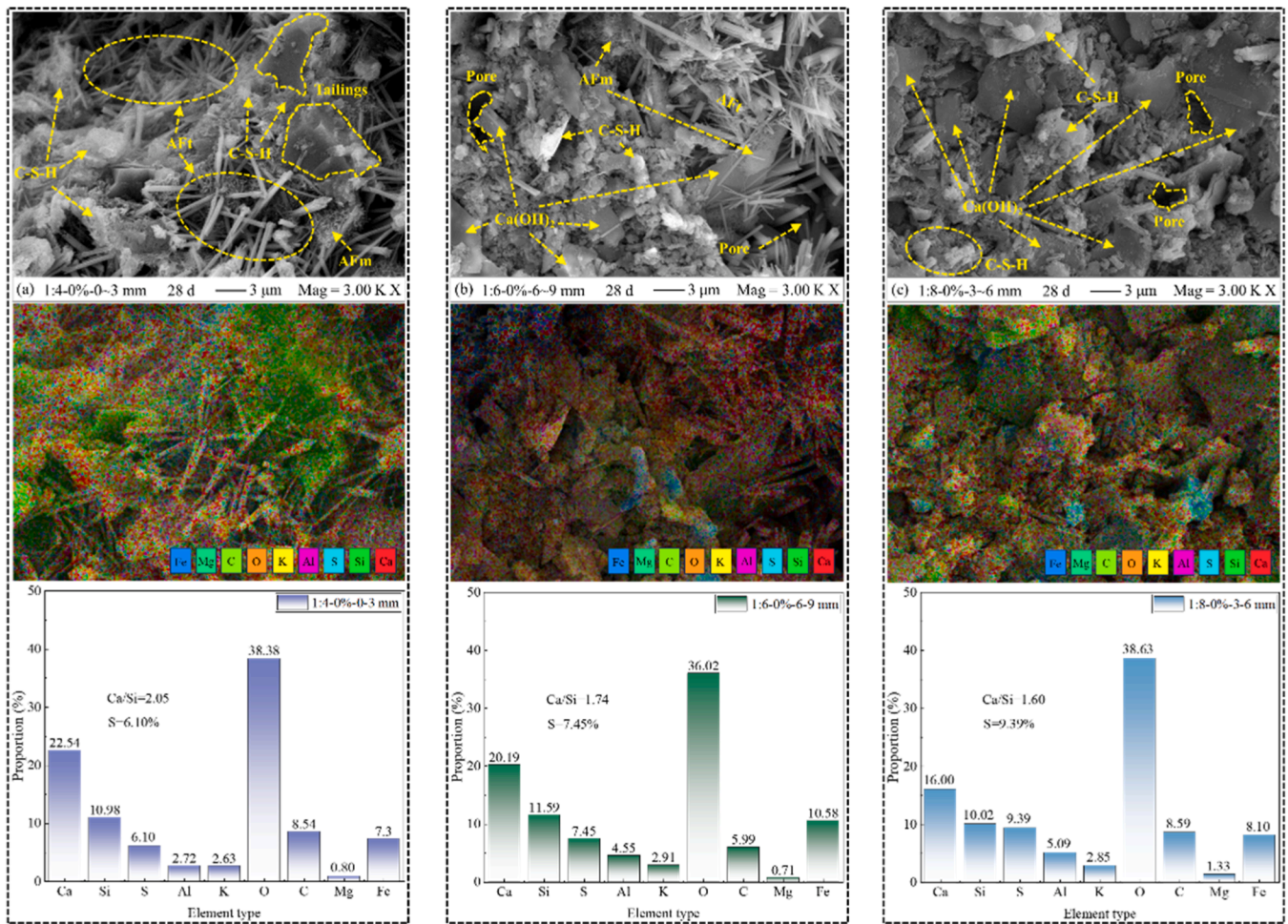


Fig. 17. SEM micrographs of 28 d cured specimens without crushed rock: (a) 1:4-0%-0-3 mm; (b) 1:6-0%-6-9 mm; (c) 1:8-0%-3-6 mm.

characteristics and macroscopic mechanical performance. For high-strength specimens (e.g., “1:4-10%-6-9 mm”), abundant Aft and C-S-H gel were observed within ITZ. Although ITZ width was fairly large, a dense, interwoven network of acicular Aft crystals effectively bridged pores and microdefects, reinforcing the weak interfacial zone and thereby contributing to the enhanced compressive strength.

Medium-UCS specimens (“1:4-40%-6-9 mm,” “1:6-30%-6-9 mm,” “1:6-50%-0-3 mm,” and “1:8-30%-3-6 mm”) contained fewer hydration products, and numerous pores and Ca(OH)_2 -enriched zones were observed. These features hindered interfacial bonding and negatively impacted the macroscopic mechanical properties. For instance, although the ITZ of “1:6-30%-6-9 mm” showed partial filling, the insufficient and unevenly distributed hydration products failed to produce a significant increase in UCS performance. The low-UCS specimen “1:8-50%-6-9 mm” exhibited a UCS value of only 0.74 MPa, far below typical backfill safety requirements. Microscopic analysis revealed that although the ITZ appeared relatively closed, extensive interconnected pores persisted, Ca(OH)_2 enrichment was prominent, and Aft and C-S-H gel were rare. This combination resulted in deprived internal compression and drastically reduced macroscopic strength.

Overall, these observations demonstrate that the content, distribution, and interlocking of hydration products within ITZ are critical determinants of the mechanical performance of CHSTCRB.

Fig. 20 presents the microstructural characteristics of CHSTCRB specimens after 56 d of curing, with emphasis on ITZ between rock aggregates and cementitious products. Measured ITZ widths for specimens “1:4-30%-0-3 mm,” “1:4-40%-6-9 mm,” “1:6-40%-3-6 mm,” “1:6-50%-0-3 mm,” “1:8-10%-0-3 mm,” and “1:8-50%-6-9 mm”

were 18.4–8.6 μm , 37.5–18.3 μm , 29.2–6.7 μm , 9.4–5.5 μm , 44.6–28.5 μm , and 17.7–15.2 μm , respectively, corresponding to UCS values of 4.49 MPa, 2.59 MPa, 2.66 MPa, 2.17 MPa, 2.45 MPa, and 0.46 MPa. Compared with the 28 d specimens, all 56 d samples exhibited a more densified ITZ structure, characterized by better pore filling, fewer interfacial voids, and significantly enhanced bonding between aggregates and the cemented matrix.

The overall increase in UCS after 56 d curing was attributed to the incorporation of crushed rock aggregates, which formed a rigid structural framework within the matrix. This coarse aggregate skeleton constrained the volumetric expansion of secondary sulfate products and impeded crack propagation, thereby effectively mitigating the mechanical degradation induced by sulfide oxidation and other long-term physicochemical reactions in high-sulfur tailings backfill. For high-UCS specimens (“1:4-30%-0-3 mm”), acicular-rod Aft crystals formed a dense, interwoven network that penetrated the ITZ, while C-S-H gel was extensively distributed within internal pores and particle gaps. This dense cementitious microstructure, coupled with the supporting aggregate skeleton, effectively filled micro-defects, enhanced interparticle bonding, and improved the macroscopic compressive performance.

Medium-UCS specimens (“1:4-40%-6-9 mm,” “1:6-40%-3-6 mm,” “1:6-50%-0-3 mm,” and “1:8-10%-0-3 mm”) exhibited larger ITZ widths, weaker contact connection, and less Aft and C-S-H gel crops compared with great-UCS specimens. Consequently, cementation was weaker and macroscopic mechanical properties were limited.

The lowest-UCS specimen, “1:8-50%-6-9 mm,” displayed pronounced microstructural defects, including numerous interconnected pores, prominent Ca(OH)_2 enrichment, and scarce Aft and C-S-H gel.

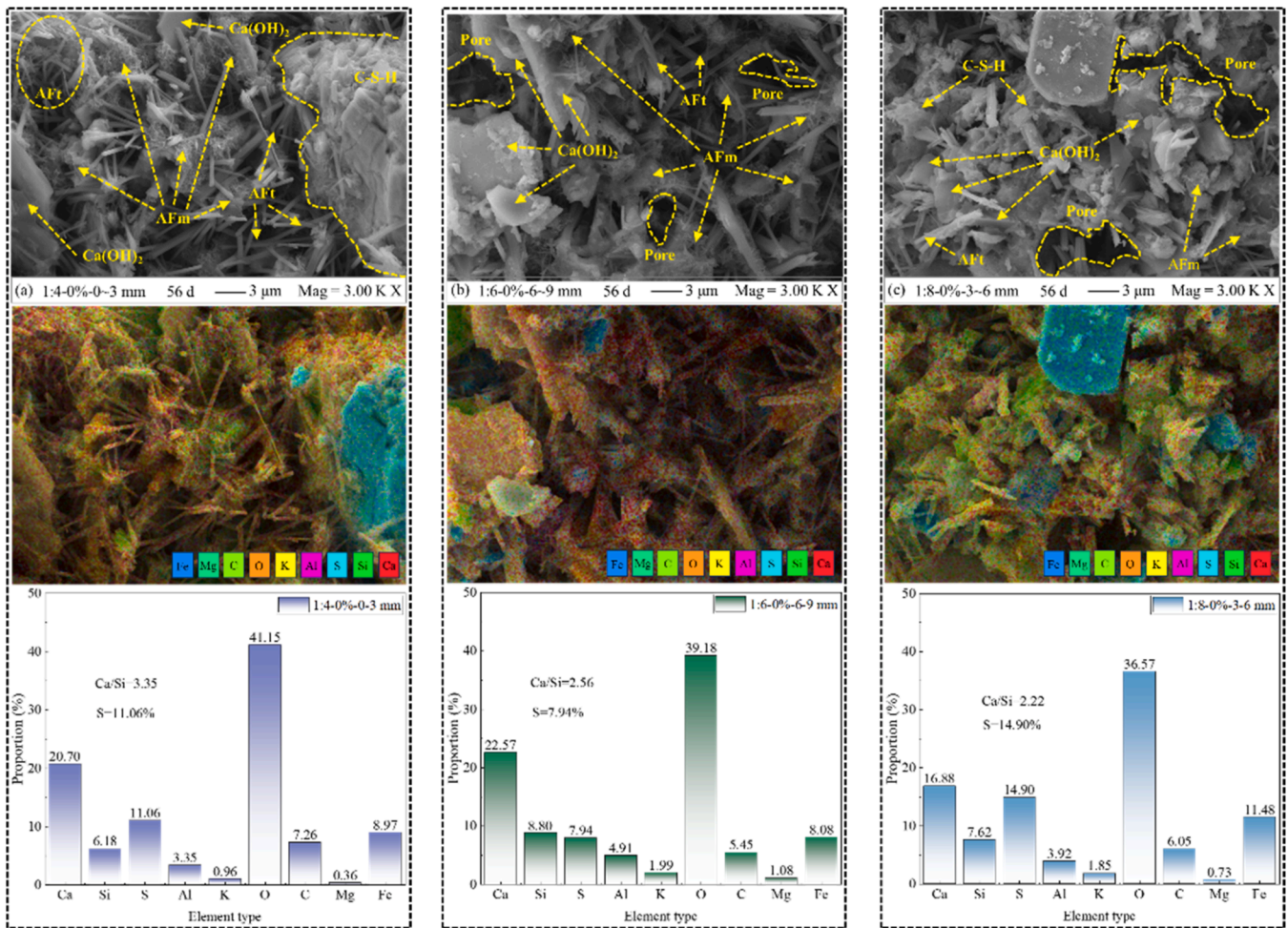


Fig. 18. SEM micrographs of 28 d cured specimens without crushed rock: (a) 1:4-0%-0-3 mm; (b) 1:6-0%-6-9 mm; (c) 1:8-0%-3-6 mm.

These features prevented effective pore filling and particle bonding, causing a weak inner formation having extremely poor compactness and very low compressive strength, insufficient for underground mine backfilling applications.

Overall, SEM observations confirm that spatial distribution, abundance, and interlocking of hydration products within the ITZ are key microstructural mechanisms governing CHSTCRB's long-term strength.

4. Conclusions

In the current study, UCS experiments and microstructure analyses were directed on specimens based on an orthogonal experimental approach. The effects of strength, high-sulfur worsening aspect, failing style, dissipated energy, and microstructural characteristics were systematically investigated. Research's leading verdicts were given below:

- (1) Orthogonal testing demonstrated that strength of CHSTCRB declined by dropping c/t ratio at both 28- and 56-day cure ages. The c/t ratio emerged as the dominant factor controlling strength, followed in influence by crushed gradation and rock content then. Properly selecting crushed rock content and gradation not only enhanced backfill strength but also mitigated the deleterious effects of high-sulfur tailings. Importantly, the combined influence of crushed rock content and gradation was synergistic, with their complementary interaction optimizing the overall mechanical performance of CHSTCRB.

- (2) Deterioration of fills with no crushed rock is governed by the modest interaction between hydrated products and pyrite oxidation, progressing through three distinct stages. Age exerts a more dominant governing influence on D_{pk} ; as curing duration rises, D_{pk} correspondingly rises, leading to a progressively looser backfill microstructure and a reduction in mechanical strength. The c/t ratio also significantly affects D_{pk} , as confirmed by microstructural observations of the specimens.
- (3) At 28- and 56-day, failure behavior of CHSTCRB was strongly dictated by crushed rock content and grain diameter, whereas c/t ratio influenced orientation of the primary fractures. Specimens with low rock content and finer grain diameters failed in a brittle tensile mode, with axial cracks traversing the entire sample. As rock content rose and particle diameter coarsened, failing evolved into a tensile-shear composite pattern, combining oblique shear cracks with axial tensile fractures. This advance was accompanied by axial stratification, where finer tailings accumulated atop coarser aggregates, and by unstable expansion fractures, highlighting internal particle rearrangement and localized structural instability.
- (4) The total energy of CHSTCRB specimens rose steadily under-loading. Elastic strain energy initially accumulated and then declined, whereas dissipated energy grew slowly at first and surged sharply in later steps. The destruction growth progressed through 4 steps: pore compaction, elastic distortion, plastic development, and post-peak collapse. In each phase, patterns of

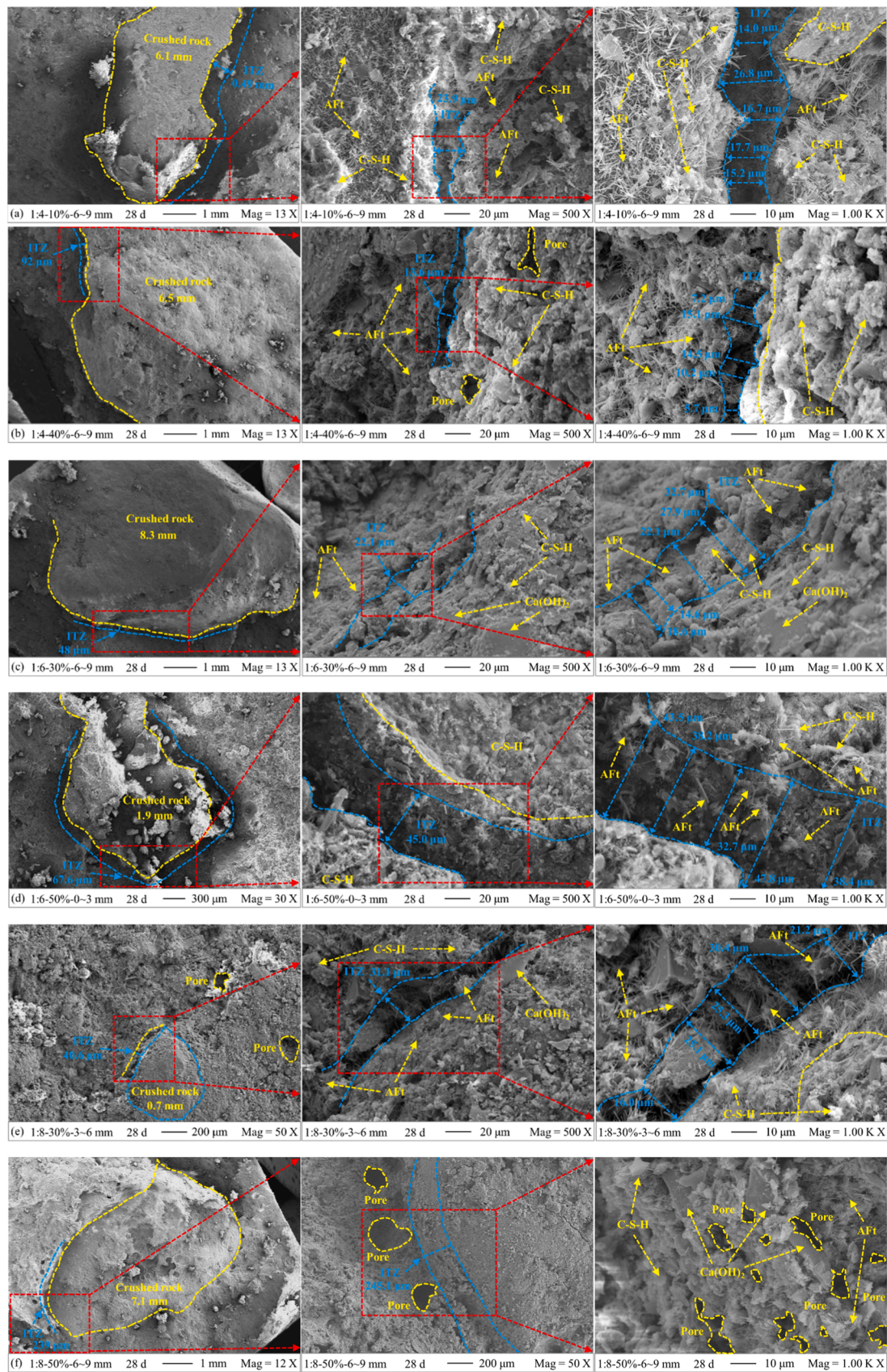


Fig. 19. Microstructure of 28-day cured CHSTCRBs: (a) 1:4-10%-6-9 mm; (b) 1:4-40%-6-9 mm; (c) 1:6-30%-6-9 mm; (d) 1:6-50%-0-3 mm; (e) 1:8-30%-3-6 mm; (f) 1:8-50%-6-9 mm.

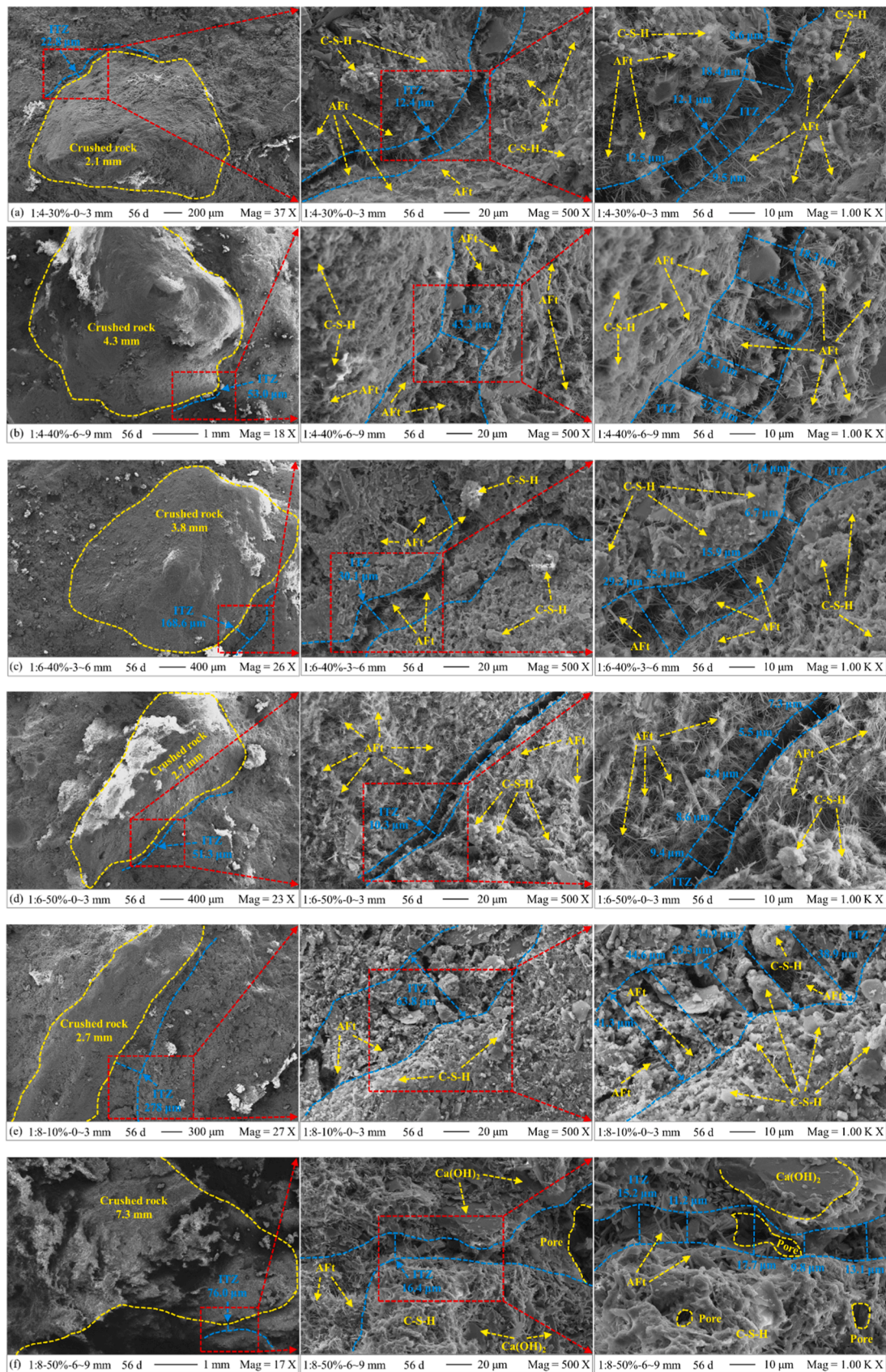


Fig. 20. Microstructure of 56-day cured CHSTCRBs: (a) 1:4-30%-0-3 mm; (b) 1:4-40%-6-9 mm; (c) 1:6-40%-3-6 mm; (d) 1:6-50%-0-3 mm; (e) 1:8-10%-0-3 mm; (f) 1:8-50%-6-9 mm.

energy evolution corresponded closely to the observed structural deformation behavior.

- (5) A higher c/t ratio promoted AFt and C-S-H formations, causing a deeper structure and improved UCS. Extended curing age further refined the ITZ and enhanced overall structural compactness. The incorporation of crushed rock aggregates mitigated the deterioration effects associated with high-sulfur tailings. In general, a narrower ITZ and greater abundance of cementitious hydration products corresponded to higher backfill strength, whereas a looser microstructure led to significant strength reduction.

CRedit authorship contribution statement

Zhiqiang Huang: Writing – original draft. **Meilin Jiang:** Investigation. **Shuai Cao:** Writing – review & editing. **Erol Yilmaz:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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