



Original Paper

Impact of fracture filling states on acid transport and conductivity in deep carbonates

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ABSTRACT

Acid fracturing is a key stimulation method for deep and ultra-deep carbonate reservoirs, where extreme temperature-stress conditions and pervasive natural fractures frequently compromise treatment effectiveness. Natural fractures act as competing flow pathways that divert and consume acid, reducing etching efficiency and limiting fracture conductivity. To address this challenge, this study establishes a geology-engineering integrated framework that links multi-scale fracture characterization with high-temperature (150 °C), high-stress (0 to 80 MPa) acidizing experiments. Geological characterization, combining 2D petrography and 3D fracture reconstruction, reveals abundant high-angle fractures with variable mineral fillings (quartz, calcite, and clay). Based on their distinct physico-chemical reactivity with acid, natural fractures are classified into three engineering-relevant types: reactive-filled fractures (RFF), inert-filled fractures (IFF), and open-conduit fractures (OCF). Correspondingly, acid transport exhibits three interference modes: deflection (RFF; controlled by coupled acid-carbonate reaction and gas effects), obstruction (IFF; dominated by siliceous mechanical blocking), and bifurcation (OCF; controlled by physical leak-off). Experimental results demonstrate that etch-induced mass loss follows OCF > IFF > RFF, whereas effective acid penetration distance follows the reverse trend (RFF > IFF > OCF), indicating that high acid consumption does not equate to effective utilization. Dolomite samples develop higher surface roughness (JRC) and show stronger sensitivity of conductivity to fracture filling type, while limestone exhibits uniformly low JRC and conductivity due to its more homogeneous dissolution. These findings clarify the mechanistic role of natural fracture fillings in acid transport and fracture conductivity and provide a quantitative basis for designing acid fracturing treatments in fractured deep and ultra-deep carbonate reservoirs.

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1. Introduction

Deep and ultra-deep carbonate formations have become key targets for future hydrocarbon development as shallow resources continue to decline (Guo et al., 2019; He et al., 2023). In China, the Tarim Basin contains some of the world's most challenging reservoirs, characterized by extreme temperature-pressure conditions, strong heterogeneity, and pervasive natural fracture-cavity

systems (Jia and Wei, 2002; Lin et al., 2015; Zhu et al., 2021). These natural fractures, formed through multiphase tectonic deformation, karstification, and diagenesis, occur across multiple scales and commonly contain mineral fillings of diverse origins (Lorenz et al., 1997; Ren et al., 2021; Zhang et al., 2023; Zeng et al., 2025). Their geometries and filling characteristics exert a first-order control on fluid transport and well stimulation performance.

Acid fracturing is the primary stimulation method for deep and ultra-deep carbonates. Unlike proppant-based treatments, its effectiveness relies on generating non-uniform etching on fracture surfaces to create stable conductive channels under high closure stress (Asadollahpour et al., 2018; Xu et al., 2021). Generally, this etching morphology and acid delivery performance are governed by the rheological properties of the acid system. For instance, cross-linked acids exhibit non-Newtonian features where shear

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thinning facilitates long-distance transport at high pumping rates, while viscoelasticity stabilizes flow structures and enhances wall-contact efficiency (Li et al., 2025a, 2025b). However, in ultra-deep formations, acid penetration remains strongly restricted by rapid reaction kinetics and diversion into natural fractures, often resulting in limited effective fracture length and rapid productivity decline (Kong et al., 2023; Gou et al., 2024; Wang et al., 2024). Although significant progress has been made in understanding acid transport and etching behavior in single-fracture systems considering these rheological complexities (Li et al., 2019; Mou et al., 2023; Yang et al., 2025), the influence of natural fractures, especially their filling states, on acid propagation remains poorly constrained.

Current studies largely treat natural fractures as geometric discontinuities or fluid-loss conduits (Li et al., 2018a, 2018b; Wu et al., 2025). Yet, field and core observations reveal that natural fractures contain fillings with widely varying mineralogy, including silica, calcite, and multiphase assemblages (Ren et al., 2020). These minerals differ drastically in both chemical reactivity with acid and mechanical behavior under stress. As a result, natural fractures may act not only as leakage pathways but also as chemical reactors, physical barriers, or flow-deflecting structures. Such coupled chemo-physical interference mechanisms have not been systematically investigated, representing a critical scientific gap that limits the accuracy of acid-fracturing design in ultra-deep formations. Meanwhile, the stimulation of ultra-deep fractured carbonate reservoirs is fundamentally constrained by temperature-stress coupling and fracture stability under high confining pressure (Jaeger et al., 2009; Ma et al., 2018). With increasing burial depth, elevated temperature and in-situ stress significantly alter rock mechanical properties, fracture aperture evolution, and conductivity degradation, making the activation and reconfiguration of pre-existing fracture systems a primary stimulation objective rather than the creation of new fractures. Classical reservoir geomechanics and stimulation theories (Fredd and Fogler, 1998; Economides and Nolte, 2000; Zoback, 2010) have demonstrated that fracture geometry, filling state, and boundary conditions play dominant roles in controlling fluid transport and treatment effectiveness in deep reservoirs. Within this theoretical framework, the experimental observations presented in this study provide laboratory-scale evidence of how different fracture-filling states interfere with acid transport and etching evolution under high-temperature conditions.

To address these questions, this study develops a geology–engineering integrated framework that links multi-scale fracture characterization with high-temperature (150 °C), high-stress (up to 80 MPa) acidizing experiments. Detailed imaging (SEM, EDS, micro-CT, and thin sections) was used to quantify fracture geometries and filling mineralogy in ultra-deep Cambrian and Ordovician carbonates. The geological basis for this fracture classification is summarized in Supplementary Material. Based on their physicochemical interactions with acid, natural fractures were systematically categorized into: (i) reactive-filled fractures (RFF), (ii) inert-filled fractures (IFF), and (iii) open-conduit fractures (OCF). A series of controlled laboratory experiments was then conducted to evaluate how these fracture types govern acid transport, etching morphology, JRC evolution, mass-loss behavior, and conductivity retention under closure stress. The results reveal three distinct interference modes—deflection (RFF), obstruction (IFF), and bifurcation (OCF)—each associated with a unique combination of chemical reactivity and physical impedance. These findings offer a mechanistic basis for predicting acid-dominant flow paths and optimizing stimulation strategies in deeply buried fractured carbonate systems.

2. Materials and methodology

2.1. Characterization methods of multi-scale natural fractures

The filling minerals within natural fractures are key determinants of their physico-chemical interactions with acid. XRD and SEM-EDS analyses (Table A1 and Figs. A1–A3) reveal that natural fractures and karst cavities in different strata of the Tarim Basin fall into two main categories: siliceous and calcareous fillings. Cast thin-section (Fig. A4) observations confirm the presence of multi-scale natural fractures with complex geometries and variable apertures, while in-situ CT imaging (Fig. A5) shows that large fractures are predominantly high-angle and commonly accompanied by dissolution fractures around karst cavities. Detailed results can be found in Supplementary Material.

Based on these findings, this laboratory model realistically reproduces the essential chemical reactivity of different filling minerals during acid–rock interactions, ensuring both geological representativeness and broad applicability of the observed experimental phenomena (see Figs. 1 and 2).

2.2. Experimental sample preparation

The laboratory experimental design was guided by the geological characteristics of ultra-deep carbonate reservoirs in the Tarim Basin. As summarized in Supplementary Material, these reservoirs are characterized by complex fracture-cavity systems with variable filling states, including open fractures, calcite-filled fractures, and siliceous-filled fractures. These geological features provide the basis for defining the four experimental fracture conditions (NTF, RFF, IFF, and OCF) investigated in this study. Although the natural fracture network is complex, this study designed a controlled, simplified laboratory model to decouple the interference mechanisms of high-angle (90° vertical) natural fractures in different filling states during acid transport, as shown in Fig. 3.

Outcrop of Cambrian dolomite and Ordovician limestone, selected for their mineralogical representative, were used as the source material for samples. These samples were machined rock plates to dimensions of 178.0 mm × 38.0 mm × 20.0 mm (±0.5 mm) following API standards. A 2 mm slot between two rock plates was used to represent an transport channel for acid transport experiments, which were designed to evaluate etched-fracture morphology and post-treatment conductivity (Tan et al., 2024). To simulate filled natural fractures, a single longitudinal slot (height: 38 mm; width: 2 mm; length: 10 mm) was sawn in each sample, and filled with particulate material sourced from rock waste. Ultimately, four samples were prepared: RFF filled with calcite, IFF filled with quartz, OCF which contained no fill material natural fracture (NTF). For the IFF and RFF samples, the fillings was emplaced and compacted within the pre-existing fracture to ensure contact with the fracture faces and to minimize internal void space.

The base acid used in this study was 20 wt% HCl, which was formulated into a cross-linked acid system using high-temperature-resistant cross-linking and stabilizing additives. The formulation was designed to enhance viscosity, improve shear stability, and retard acid–rock reaction under elevated temperature conditions representative of ultra-deep reservoirs. The cross-linked acid fluid used in this study follows the formulation applied in the Shunbei Oilfield, Tarim Basin. It contains 20 wt% HCl, 1% thickener, 4% corrosion inhibitor (a mixture of inhibitor A and B), 1% iron stabilizer, 1% demulsifier, 0.2% conditioner, and 1.2% cross-linking agent (A:B = 1:1).

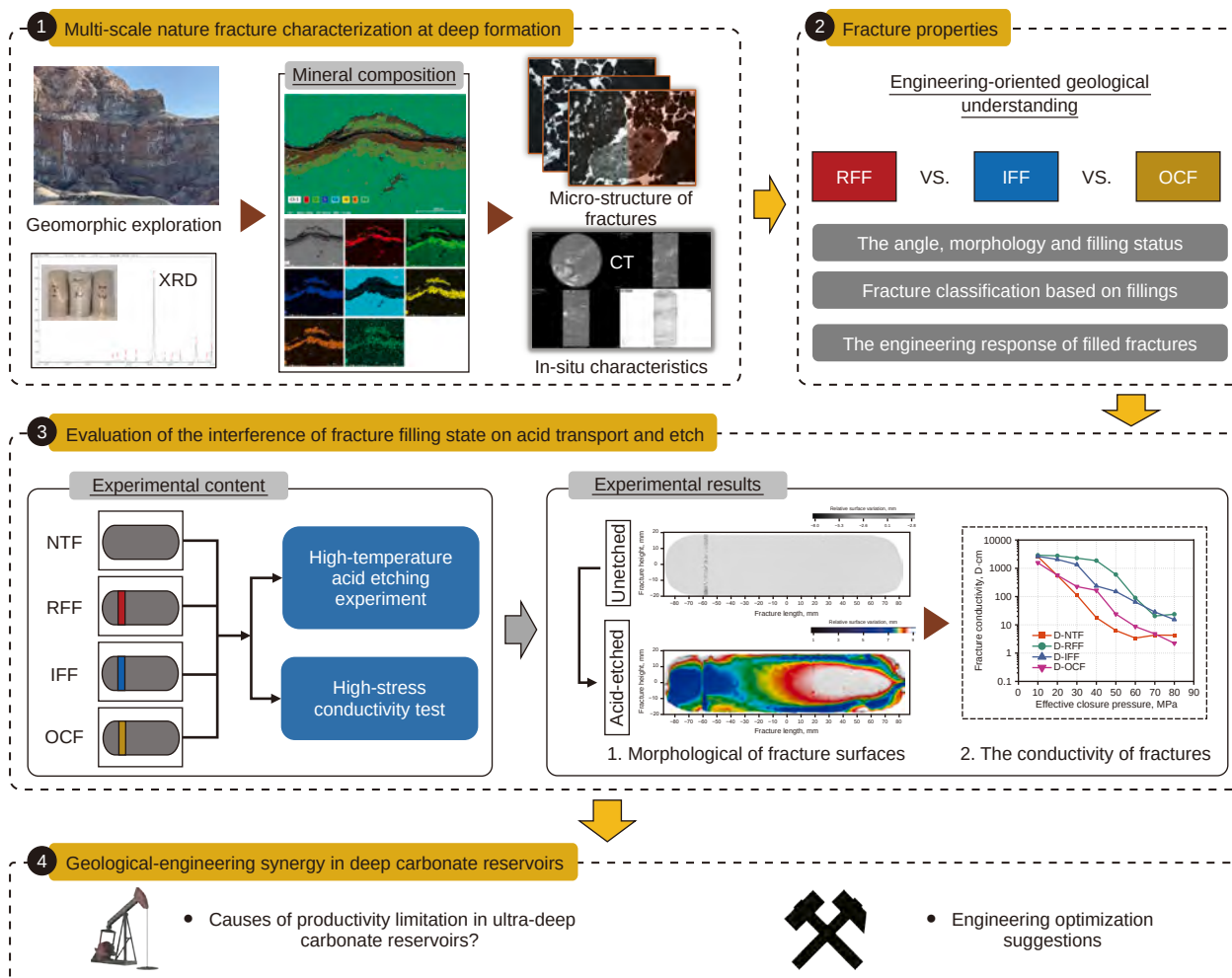


Fig. 1. Integrated geological and experimental framework.

2.3. Experimental setup and scheme

2.3.1. Experimental setup

The experimental apparatus was designed to simulate high-temperature acid–rock interactions and assess post-etching fracture performance, as shown in Fig. 4. It consists of three modules: (1) a high-temperature acid-etching unit to simulate acid transport and reaction within fractures; (2) a surface scanning system for quantitative characterization of etched fracture morphology; and (3) a conductivity test device to evaluate fracture conductivity under high closure stress (see Fig. 5).

In the deep and ultra-deep carbonate formations of the Tarim Basin, high temperature ($\geq 150\text{ }^{\circ}\text{C}$) and high closure stress conditions ($\geq 60\text{ MPa}$) are prevalent. Under these conditions, eight sets of acidizing and conductivity experiments were conducted. The experimental procedures was as follows:

- (1) Filled fracture: A 2 mm-wide groove was cut perpendicular to the rock plate surface, 30 mm from the acid entry point, and filled with minerals to simulate natural fractures encountered during acid transport.
- (2) Flow channel: A 2 mm-wide gap was maintained between the two rock plates.
- (3) High-temperature acid etching: The outlet pressure was maintained at 3 MPa using a back-pressure regulator to simulate the flowing pressure within fracture. The device and rock plates was preheated to $150\text{ }^{\circ}\text{C}$ for 60 min, followed

by acid injection at 30 mL/min for 50 min to etch the rock plate, creating acid-etched fractures.

- (4) Morphology characterization: The acid-etched fracture were digitally visualized to capture acid-etched fracture changes.
- (5) High-stress conductivity testing: Under closure stresses ranging from 10 to 80 MPa, the conductivity of acid-etched fracture was measured. The test device was held at each stress level for 30 min, and average conductivity was recorded.

2.3.2. Experimental scheme

In this study, eight experimental groups were designed to compare the interference effects of four types of filled fractures on acid transport within the main fracture under different lithologies. The detailed experimental scheme is presented in the following Table 1.

3. Results

3.1. Evidence of diverse natural fracture fillings and proposed classification

The Lower Cambrian Xiaerbulake Formation is characterized by high-angle, bed-crossing structural fractures and exhibits the strongest diagenetic overprint among the studied strata,

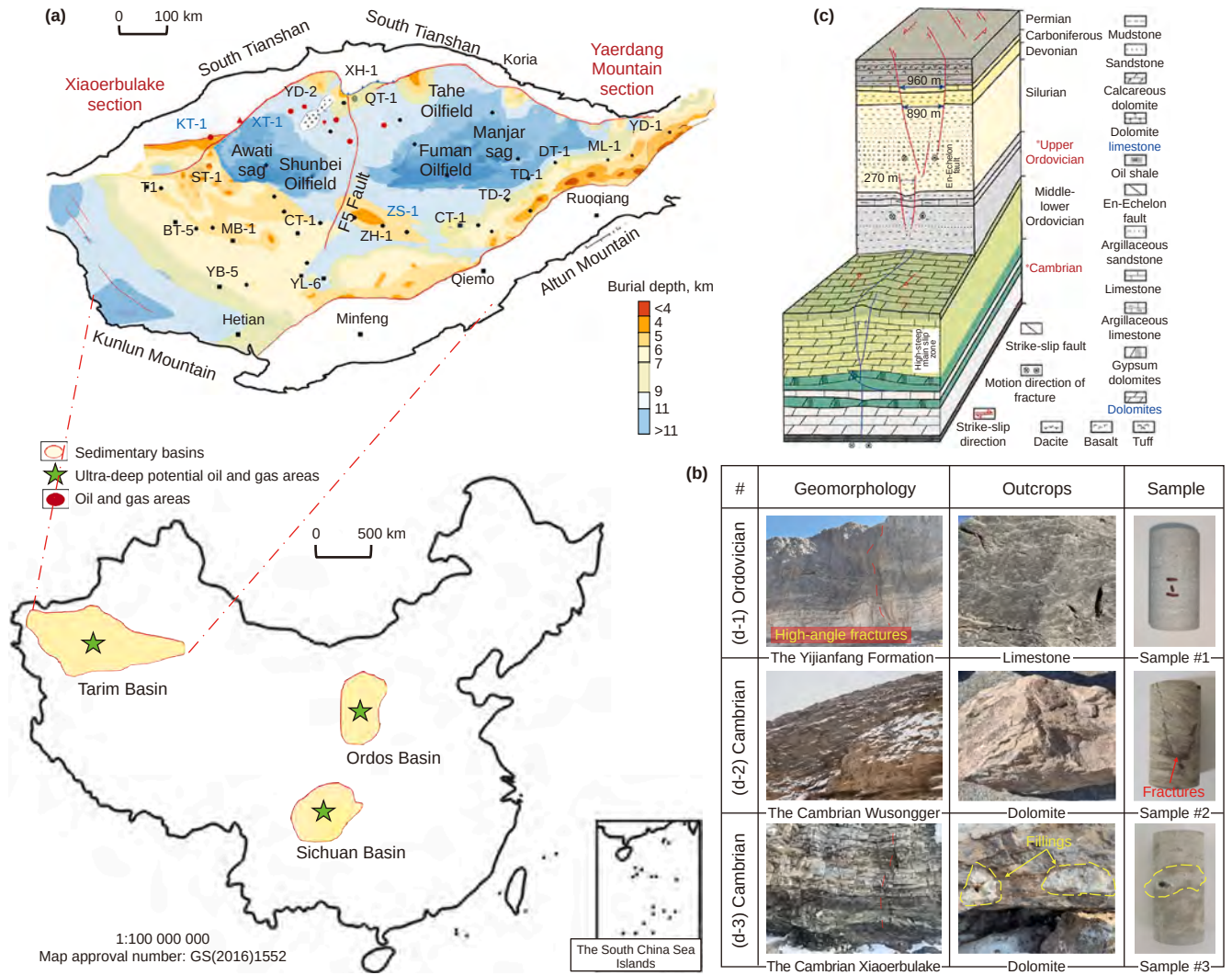


Fig. 2. The geological framework of ultra-deep strata in the Tarim Basin. (a) Burial depth of oil and gas, (b) distribution of the three major ultra-deep oil and gas basins in China, (c) vertical stratification and planar segmentation of the Tarim Basin, (d) geological characteristics and multi-scale fracture features of the sampled carbonate formations from the Tarim Basin (modified from (Ma et al., 2022; Wang et al., 2021; Su et al., 2025)).

consistent with its greatest burial depth. A fracture-cavity system developed through tectonic fracturing followed by hydrothermal karstification is observed. Fractures and dissolution cavities are partially filled with calcite, indicating incomplete mineral filling after fracturing. The Cambrian Wusongger Formation shows pervasive dolomitization and siliceous replacement. Natural fractures within this formation contain multi-phase fillings dominated by siliceous minerals. Irregular fracture geometries and dissolution features along fracture margins are commonly observed. In contrast, the shallower Ordovician Yijianfang Formation exhibits partial fracture filling and compaction-related fracture closure. Due to its relatively simple diagenetic evolution, a considerable proportion of fractures remain open, with structural fractures and dissolution cavities constituting the primary storage spaces. Overall, natural fractures in these ultra-deep carbonate formations are controlled by tectonic deformation, karstification, and diagenetic modification (Fig. 6). The resulting fractures display variable apertures and are predominantly filled with calcareous or siliceous minerals. Experimental results are provided in Supplementary Material. Based on mineralogical composition, natural fractures were classified according to their dominant fillings and their expected responses to acid exposure. Fractures dominated by

siliceous fillings were defined as IFF, in which the filling minerals exhibit negligible reactivity with acid. Fractures filled predominantly with calcite were classified as RFF, where the fillings readily dissolve upon acid contact. Unfilled fractures, commonly observed in the Ordovician strata, were identified as OCF and are characterized by open apertures. In addition, intact samples without pre-existing fractures were designated as the NTF. This classification distinguishes fracture types with different mineralogical states and geometric characteristics relevant to subsequent experimental observations.

3.2. Rheological characteristics

Gelled acid, cross-linked acid, and solid acid are widely used in acidizing treatments for production enhancement in ultra-deep carbonate formations (Liu et al., 2023; Guo et al., 2025). Table 2 below shows the formulations and specific compositions of three acid fracturing fluid, and Fig. 7(a–c) display the physical appearance of three acid fracturing fluid systems employed in the Tarim Basin: cross-linked acid, gelled acid, and solid acid. The rheological profiles of these fluids, measured at 150 °C under a constant shear rate of 170 s⁻¹ for 80 min, are presented in Fig. 7(d–f). The results show

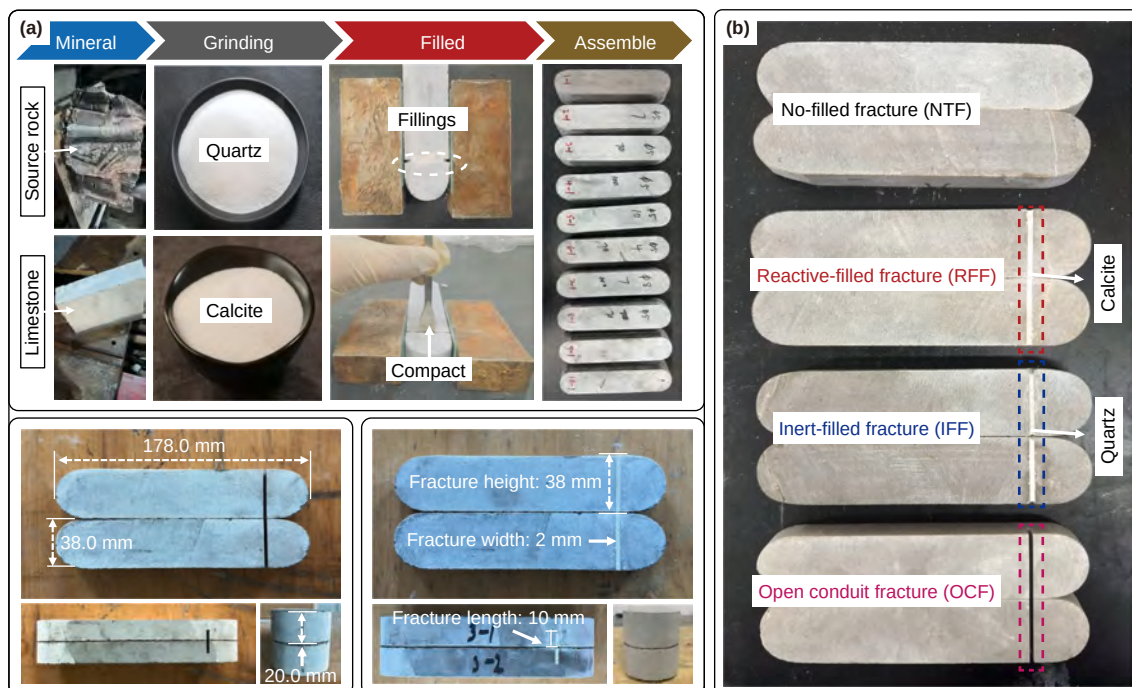


Fig. 3. Preparation process of experimental samples with different fracture filling types. (a) Sample preparation of fractures with filling materials, (b) pre-existing fractures of four type, NTF (no-filled), RFF (calcite), IFF (quartz), and OCF (unfilled).

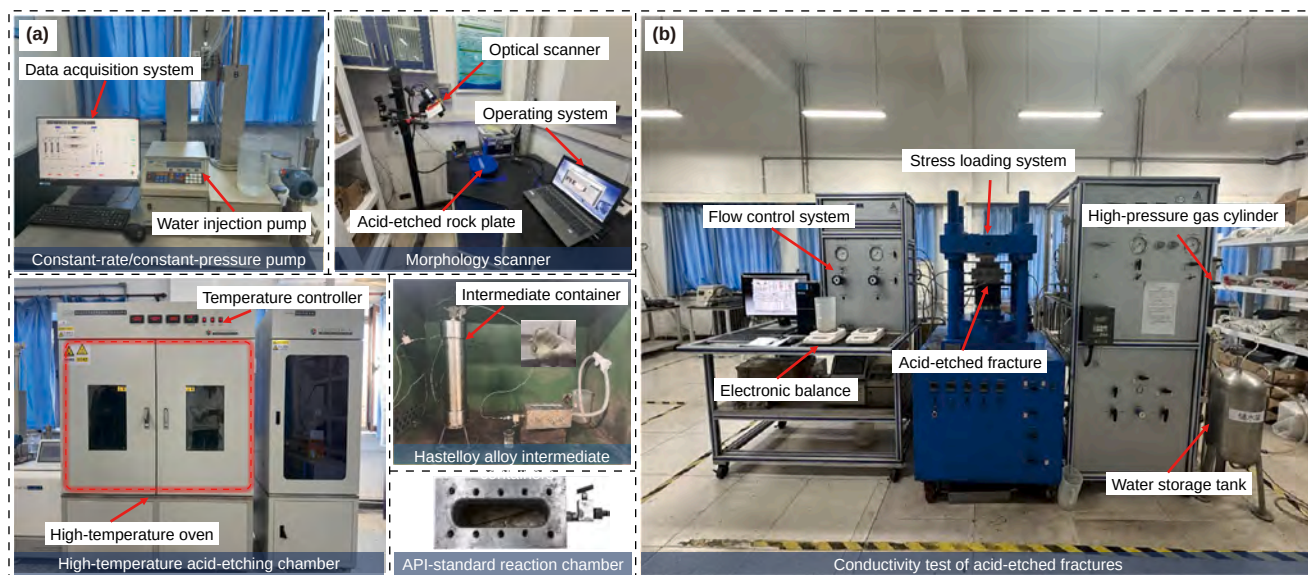


Fig. 4. Integrated laboratory system for evaluation of acid fracturing. (a) Acidification simulation experimental device, (b) conductivity testing device.

that the three acid systems exhibit distinct viscosity responses to continuous shearing under varying temperature conditions. At room temperature (23–25 °C), the apparent viscosities of the cross-linked acid, gelled acid, and solid acid were 122.70, 78.36, and 57.64 mPa·s, respectively. During the initial temperature ramp from 25 to 150 °C within 10 min, all systems experienced a pronounced decrease in apparent viscosity. After the temperature stabilized at 150 °C, the apparent viscosities remained relatively stable despite continued shearing, fluctuating within a narrow range. Under sustained high-temperature conditions (150 °C), the average apparent viscosity of the cross-linked acid was 59.48 mPa·s, compared with 33.40 mPa·s for the gelled acid and 16.50 mPa·s for

the solid acid. Among the three systems, the cross-linked acid maintained the highest viscosity, exceeding that of the gelled acid by 26.08 mPa·s and that of the solid acid by 42.98 mPa·s.

Fig. 7(g–i) present the variations in viscosity and acid concentration of the cross-linked acid during the acidizing experiments. It should be noted that viscosity measurements were performed on the effluent fluids collected at the outlet, representing the bulk rheological properties of the acid systems after fracture-scale flow and reaction, rather than the local in-fracture viscosity. As shown in Fig. 7(g), when the temperature increased to 150 °C, the viscosity of the fresh acid decreased markedly. At this stage, neither acid-rock reaction nor shearing of the rock plate occurred. Minor

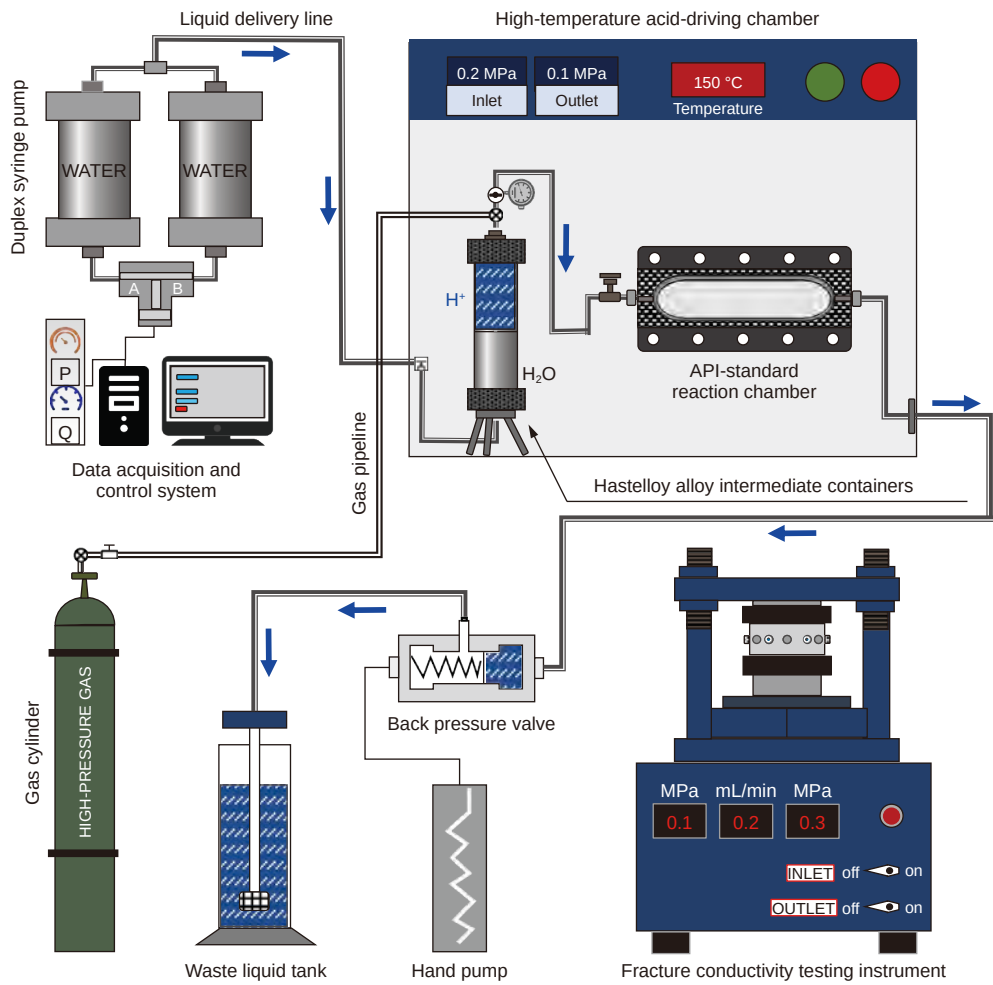


Fig. 5. Schematic diagrams of full acid fracturing process.

Table 1
Experimental schemes for evaluating the role of filled fracture in acidizing-induced productivity enhancement.

Sample	Matrix lithology	Filling	Categories	Fracture width, mm	Dip angle, °
#D-1	Dolomite	\	NTF	2	90
#D-2	Dolomite	Calcite	RFF	2	90
#D-3	Dolomite	Quartz	IFF	2	90
#D-4	Dolomite	\	OCF	2	90
#C-5	Limestone	\	NTF	2	90
#C-6	Limestone	Calcite	RFF	2	90
#C-7	Limestone	Quartz	IFF	2	90
#C-8	Limestone	\	OCF	2	90

differences in initial viscosity values were observed among tests, which are attributed to preparation variability; however, the magnitude of these differences was small and did not affect comparative analysis. Across the four acidizing experiments involving filled fractures, the viscosity of the spent acid remained close to that of the fresh acid, with absolute reductions ranging from 0 to 6 mPa·s.

Fig. 7(h) and (i) show the concentration variations of fresh and spent cross-linked acid for two rock types under different fracture-filling conditions. During a 50 min injection at a flow rate of 30 mL/min, the concentration of fresh acid remained constant at 20%, whereas the spent acid concentration varied with both lithology

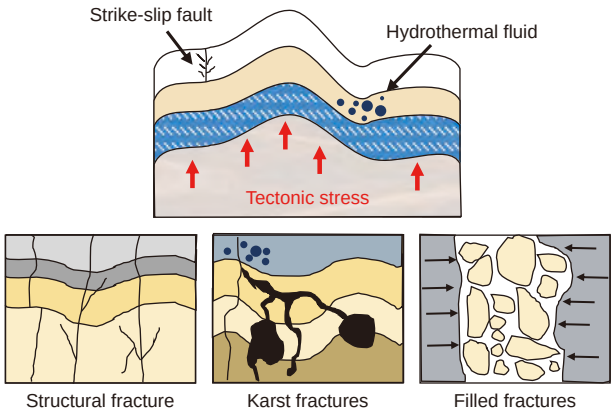


Fig. 6. Schematic illustration of the formation and evolution models for different filled types of natural fractures and cavities in carbonate stratas.

and fracture type. For dolomite, the spent acid concentrations corresponding to NTF, RFF, IFF, and OCF were 18.69%, 17.87%, 17.25%, and 16.79%, respectively, with absolute reductions of 1.31%, 2.13%, 2.75%, and 3.21%. For limestone, the corresponding spent concentrations were 18.62%, 17.89%, 17.38%, and 16.43%, yielding absolute reductions of 1.38%, 2.11%, 2.62%, and 3.57%. Overall, in both lithologies, the concentration loss of the acid solution was

Table 2
Formulas of acid systems used in shear test.

Acid fluid	Formula
Cross-linked acid	20 wt% HCl + 1% thickener + 4% corrosion inhibitor (a mixture of inhibitor A and B) + 1% iron stabilizer + 1% demulsifier + 0.2% conditioner + 1.2% cross-linking agent (A:B = 1:1)
Gelled acid	20 wt% HCl + 1.5% corrosion inhibitor + 0.15% citric acid + 0.5% acidizing fluid-discharging aid + 0.55% acid thickener + 0.5% acidizing foaming agent
Solid acid	20% acid ester + 0.8% thickening agent + 1.0% corrosion inhibitor + 1.0% fluid-discharging aid + 0.1% gel breaker + 1.0% iron-ion stabilizer

limited, with absolute reductions ranging from 1.31% to 3.57%. Measurements of acid concentration before and after acid etching show no significant reduction in acid strength over the experimental duration. Similarly, the apparent viscosity of the acid fluid remains relatively stable before and after the etching experiments, with no abrupt degradation observed. The real-time rheological behavior of the acid during transport within fractures is difficult to be directly captured. However, the relatively small variations in viscosity and acid concentration provide indirect evidence of the structural integrity of the acid system. The experimental results indicate that the cross-linked acid maintains stable structural integrity under high-temperature conditions, with only limited permanent thermal degradation observed. These results indicate that the acid-fluid exhibited good bulk rheological stability after fracture-scale flow.

3.3. Morphology of acid-etched fractures under different interference modes

The fracture configurations investigated in the laboratory are representative of the dominant natural fracture types observed in ultra-deep carbonate reservoirs of the Tarim Basin (see Supplementary Material), ensuring that the experimental conditions are geologically relevant. By digitizing the fracture surfaces before and after acid etching, the spatial distribution of relative etching features and acid transport patterns was visualized. The color scale represents relative height variations derived from surface morphology reconstruction. Cool colors (blue) indicate regions with relatively enhanced etching features, warm colors (green to red to yellow) represent areas with weaker etching features, and white denotes under-etched regions. Based on the color maps, the acid-etched fracture surfaces are divided into an etched zone (cool colors), a transition zone (warm colors), and an under-etched zone (white). The etched zone indicates the dominant acid transport pathway and the effective penetration extent.

For the NTF case in Cambrian dolomite (Fig. 8(a)), heterogeneous etching features are observed. Groove-like etched regions develop along the fracture surface, whereas insoluble components remain as topographic protrusions after acid exposure. As a result, etched zones are mainly concentrated near the acid entry region and in the lower portions of the fracture surface. In the presence of RFF (Fig. 8(b)), distinct etching features appear at the intersection between the acid-etched fracture and the filled fracture, indicating dissolution of the fracture fillings. Two narrow, strip-shaped etched zones are observed along the upper and lower

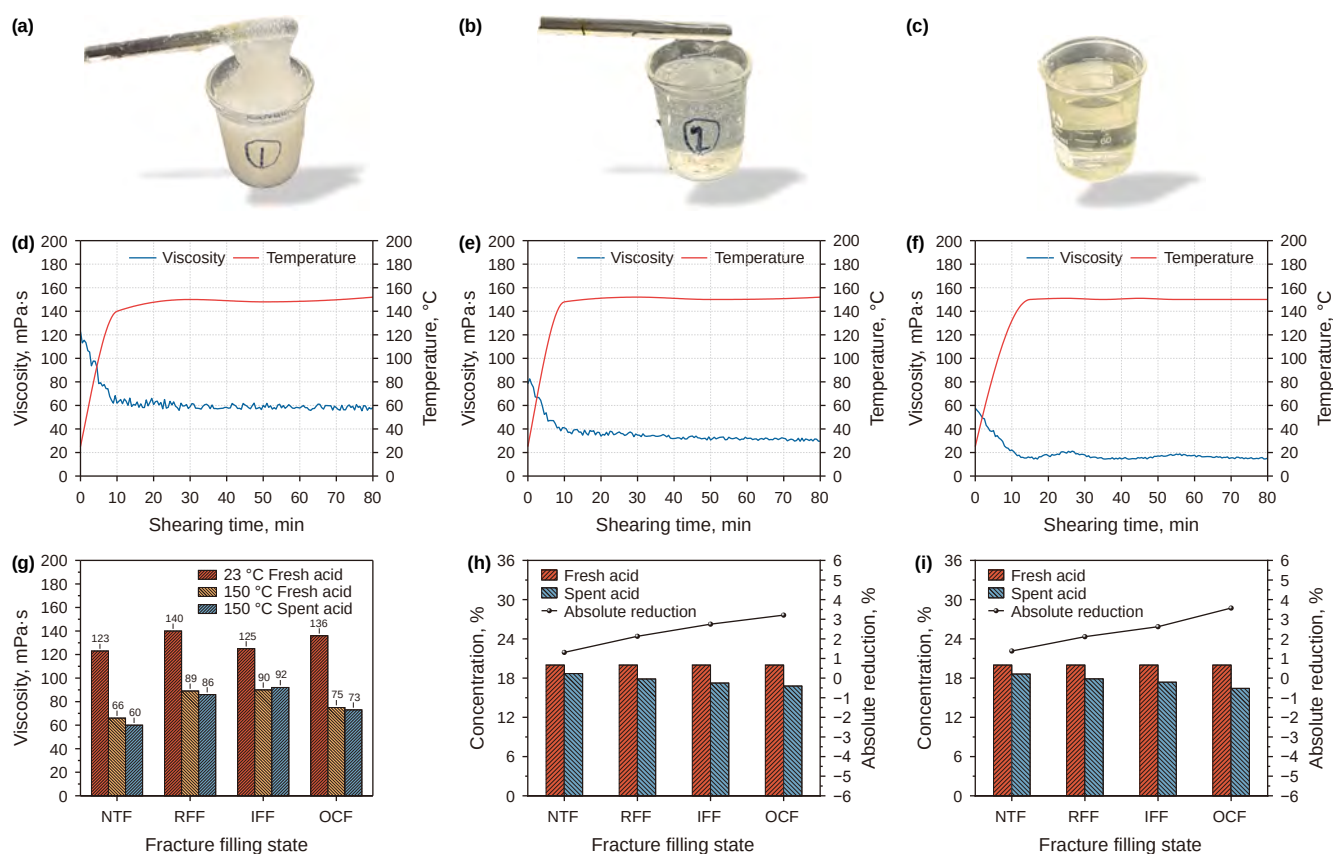


Fig. 7. Rheological behavior of representative acid systems under continuous shear at elevated temperature. (a–c) Physical appearance of cross-linked acid, gelled acid, and solid acid, respectively, (d–f) apparent viscosity evolution of cross-linked acid, gelled acid, and solid acid as a function of shear time, (g) comparison of apparent viscosities between fresh and spent cross-linked acid during acidizing experiments, (h) acid concentration evolution during dolomite acidizing experiments and (i) acid concentration evolution during limestone acidizing experiments.

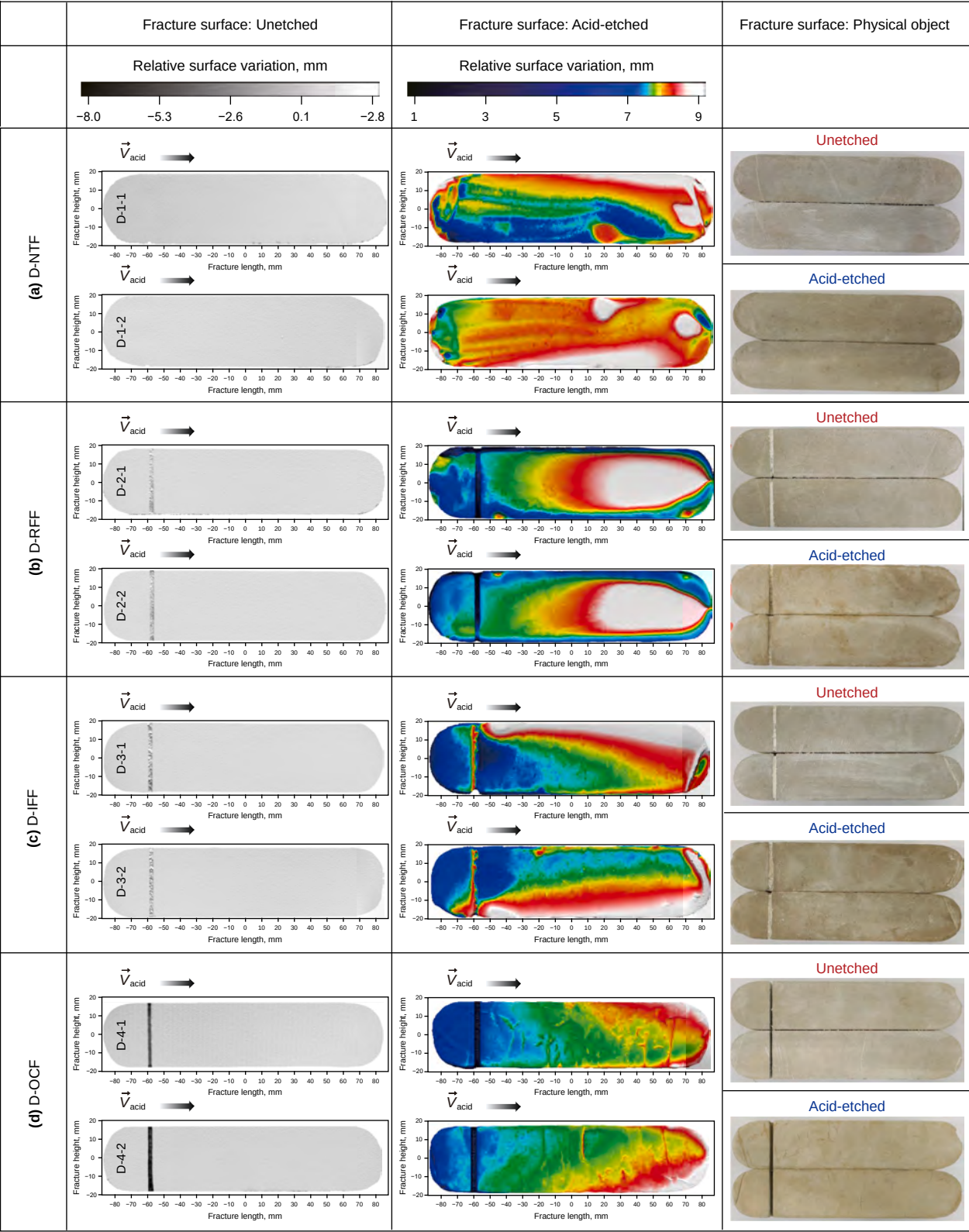


Fig. 8. Digital and physical images of dolomite rock slabs surfaces before and after acid etching. (a) Acid etching on dolomite without the natural fractures, (b) acid etching on dolomite with RFF, (c) acid etching on dolomite with the IFF, (d) acid etching on dolomite with OCF.

boundaries of the fracture surface, forming approximately parallel bands. The area between these bands corresponds to the transition and under-etched zones, which exhibit an ellipsoidal morphology

with a relatively smooth leading edge and a sharper trailing edge, occupying most of the fracture surface. When IFF is present (Fig. 8(c)), the fracture surface shows a weak response to acid

etching. Insoluble fillings persist as protrusions after etching, limiting the development of etched zones to the upstream and downstream sides of the IFF. Consequently, the effective penetration extent of acid etching is restricted, and the downstream portion of the fracture surface is dominated by the transition zone. For the OCF case (Fig. 8(d)), etching features are concentrated at the intersection between the OCF and the acid-etched fracture. The upstream side of the intersection is characterized by the etched zone, whereas the downstream side corresponds primarily to the transition zone. As the acid propagates along the fracture surface, part of the acid simultaneously enters the OCF, producing a bifurcated etching pattern. Overall, the effective penetration extent of acid etching follows the order $RFF > IFF > OCF$, indicating that the presence and filling state of natural fractures exert a strong control on the spatial distribution and relative extent of acid etching.

In the high-temperature acid-etch experiments on limestone, the overall etching patterns are generally similar to those observed in dolomite but exhibit lower spatial heterogeneity. For the NTF case (Fig. 9(a)), the etched zone remains concentrated near the lower portion of the fracture surface; however, groove-like etching features are absent. Both the vertical extent of the etched zone and the lateral width of the transition zone between etched and under-etched regions are reduced. Acid transport along the fracture surface is predominantly unidirectional, and vertical development of etching features is limited. In the presence of RFF (Fig. 9(b)), two strip-shaped etched zones develop along the fracture surface. Compared with dolomite, these etched zones occupy a larger proportion of the fracture surface, accompanied by a reduction in the extent of the transition and under-etched zones between them. When IFF is present (Fig. 9(c)), inert fillings impede forward acid transport, resulting in etched zones primarily distributed on the upstream and downstream sides of the IFF. For limestone samples, the downstream etched zone is more pronounced. In the OCF case (Fig. 9(d)), etching features are concentrated at the intersection between the OCF and the acid-etched fracture. Although differences exist between the etching patterns of limestone and dolomite, the effective penetration extent of acid etching under filled-fracture conditions follows the same trend in both lithologies, namely $RFF > IFF > OCF$.

To quantify the acid etching effect on the rock plates, a digital evaluation method was adopted (Li and Zhang, 2015). By establishing a coordinate system on the digital images of the acid-etched fracture, the joint roughness coefficient (JRC) of the acid-etched fracture under four filling conditions was calculated. The surface profile was determined using the following equation:

$$JRC = 4.4192R^{0.6482} \quad (1)$$

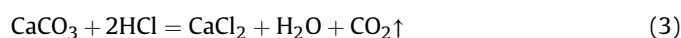
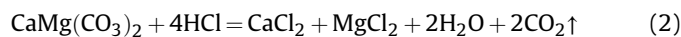
The joint roughness of acid-etched fractures is denoted as JRC, where 0.6482 is the correction coefficient specific to acid-etched surfaces, and R represents the root mean square of the first derivative of the fracture profile. Although the empirical relation of JRC was originally developed for quantifying the roughness of mechanically generated fracture surfaces, several studies (Wang et al., 2023; Gou et al., 2025) have shown that the statistical correlation between profile parameter R and apparent roughness can also be applied as a comparative indicator for chemically etched surfaces. Acid-etched fractures differ from mechanical fractures in their formation mechanisms; however, both processes produce surface asperities that can be effectively captured through profile-based descriptors. In this study, JRC is therefore used not as an absolute roughness parameter, but as a relative roughness index to compare the etching intensity among different interference modes, as shown in Fig. 10.

In the dolomite acid etching experiments (Fig. 11(a) and (b)), the presence of filled fractures increased the JRC values. When IFF was present, the JRC reached highest level, with a median value exceeding 7 and the widest distribution range. In contrast, the NTF yielded the lowest JRC, with a median around 4.5 and a relatively narrow distribution. Both IFF and OCF showed similar effects, with median JRC values near 5.8 and intermediate distribution ranges. In limestone acid etching experiments (Fig. 11(c) and (d)), the JRC values for limestone were generally lower than those for dolomite. Although filled fractures caused a slight increase in JRC, the overall JRC values across the four filling types remained similar, ranging from 4.5 to 6, with concentrated distributions.

The microscopic acid-etching characteristics were examined using SEM (Fig. 12). Distinct differences in particle morphology and etching features are observed between limestone and dolomite. Prior to acid etching, limestone exhibits intact calcite crystal faces with dense packing (Fig. 12(a)). The carbonate phase is relatively pure and free of visible impurities (Fig. 12(b)), and only a limited number of microfractures are present, with a localized spatial distribution. After acid etching, the limestone crystal surfaces become generally flattened (Fig. 12(d) and (e)). Although local roughness develops in some areas (Fig. 12(f)), the crystals remain closely arranged, and no large-scale penetrating dissolution grooves or pronounced hierarchical surface undulations are observed. In contrast, dolomite shows markedly different microscopic features. Before acid etching, clear crystal boundaries are present (Fig. 12(g)), with coarser and more irregular crystal grains accompanied by non-carbonate impurity phases (Table A1 and Fig. 12(i)). After acid etching, selective dissolution is evident. Strip-like rough structures and relatively large acid-etch pores develop preferentially along crystal boundaries, pre-existing defects, and impurity-rich zones (Fig. 12(k) and (l)).

3.4. Acid-rock reaction kinetics

During acid flow, chemical reactions between the acid and the fracture surface result in overall rock mass loss, reflecting the integrated outcome of acid-rock interaction. To quantify this global reaction response, the masses of both rock plates in each experimental group were measured before and after acid etching. The paired plates within each group exhibited comparable mass losses, indicating similar acid consumption behavior under identical injection condition. Importantly, the measured mass loss represents the combined effects of acid-rock reaction kinetics, effective contact area, and local residence time. Therefore, mass loss is used in this study to characterize the overall extent of acid-rock interaction under different fracture configurations, while the identification of interference modes is based primarily on spatial etching patterns, pressure response characteristics, and fracture conductivity evolution. The acid-rock reaction equations for dolomite and limestone are expressed as follows:



Experimental results show that the presence of filled fractures leads to increased overall mass loss during acid etching of dolomite at high temperature (Fig. 13). The measured mass loss varies systematically among different fracture configurations. The NTF case exhibits the lowest mass loss (7.66%) and is taken as the reference condition. Compared with NTF, the OCF configuration shows the largest mass loss (19.50%, corresponding to an increase

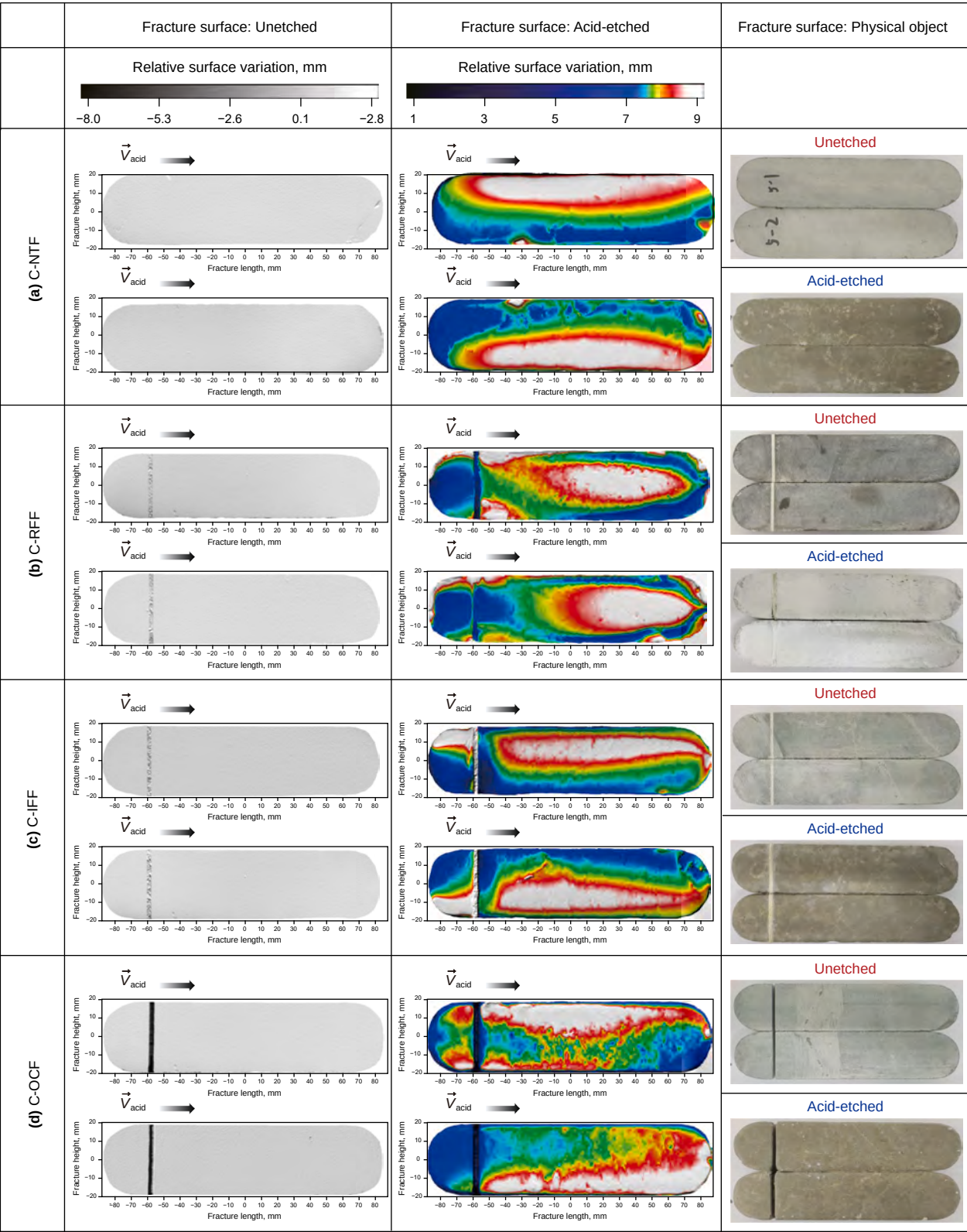


Fig. 9. Digital and physical images of limestone rock slabs surfaces before and after acid etching. (a) Acid etching on limestone without the natural fractures, (b) acid etching on limestone with RFF, (c) acid etching on limestone with the IFF, (d) acid etching on limestone with OCF.

of 11.84%), followed by the IFF case (15.53%, +7.87%) and the RFF case (12.02%, +4.36%). Overall, the total mass loss follows the order OCF > IFF > RFF > NTF.

Similar to dolomite, the presence of filled fractures also leads to increased overall mass loss during acid etching of limestone (Fig. 14). The measured mass loss under different fracture

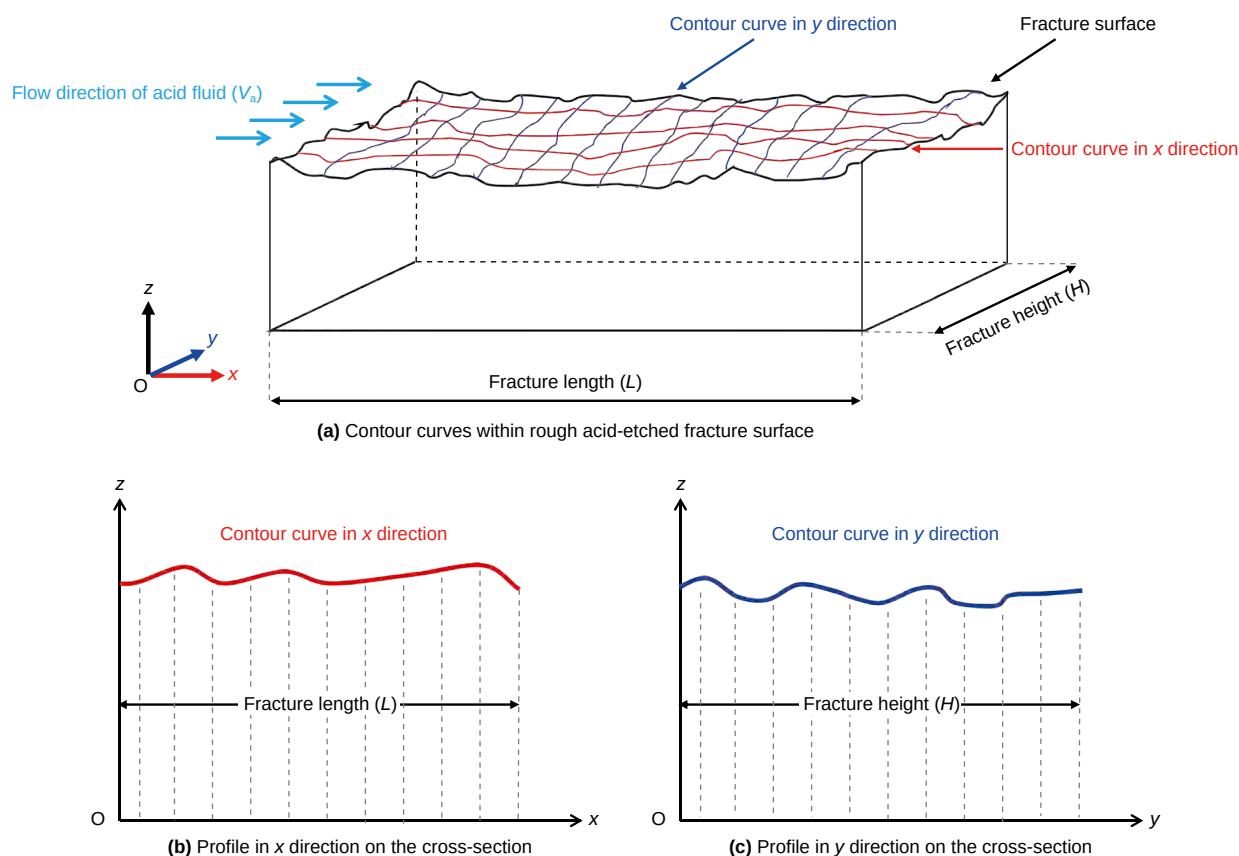


Fig. 10. Schematic diagram of rough surface contour curve.

conditions is 9.51% for NTF, 14.52% for RFF (+5.01%), 17.31% for IFF (+7.80%), and 23.73% for OCF (+14.22%). The ordering of mass loss among different fracture configurations is consistent with that observed in dolomite, following the sequence $OCF > IFF > RFF > NTF$. Across all fracture conditions, the absolute mass loss measured for limestone is higher than that for dolomite. However, the differences in mass loss between the two lithologies under the same fracture condition remain relatively limited, with increases of 1.85% (NTF), 2.50% (RFF), 1.78% (IFF), and 4.23% (OCF), respectively. These differences reflect the combined effects of mineralogical composition and acid-rock reaction characteristics. According to the reaction stoichiometry (Eqs. (2) and (3)), calcite consumes fewer moles of acid per mole of mineral than dolomite, which contributes to a slightly higher overall mass loss for limestone under identical acid injection conditions.

3.5. Conductivity of acid-etched fractures under high closure stress

Fracture conductivity tests were performed after acid etching at 150 °C. Conductivity measurements were conducted at ambient temperature (25 °C) under progressively increasing closure stress for four fracture conditions (NTF, RFF, IFF, and OCF), as shown in Fig. 15. For both dolomite and limestone samples, fracture conductivity decreases monotonically with increasing closure stress. A pronounced reduction is observed between 20 and 60 MPa, while the rate of conductivity loss becomes less sensitive at higher stress levels. At closure stresses exceeding 60 MPa, the measured conductivity approaches near-zero values for most cases.

In dolomite, conductivity exhibits variation across different fill conditions. The RFF and IFF conditions maintained the highest conductivity, followed by OCF and NTF. Notably, under high closure

stress (>60 MPa), RFF and IFF samples still preserved conductivity in the range of 10–100 D-cm, outperforming OCF and NTF (<10 D-cm). Under low stress (10–40 MPa), RFF and IFF samples demonstrated stable conductivity without abrupt decrease. On the other hand, limestone exhibited a different conductivity. When the closure stress was below 30 MPa, the RFF and NTF cases had higher conductivity than IFF and OCF. However, as stress exceeded 30 MPa, the differences among all cases narrowed. Although NTF and RFF initially showed greater conductivity, their decrease was rapid. Conversely, IFF and OCF began with lower conductivity but exhibited slower reduction. At stresses above 60 MPa, conductivity in all acid-etched fracture of limestone cases dropped to near-negligible levels. Comparatively, acid-etch fracture of dolomite consistently exhibited higher conductivity than limestone under identical stress conditions. Additionally, the impact of filled fractures on conductivity was more significant in dolomite. While the RFF condition in dolomite generally outperformed other groups, but all acid-etched fractures showed conductivity below 20 D-cm at 80 MPa.

The conductivity of the acid-etched fracture is governed by surface protrusions, which provide mechanical support to maintain the fracture aperture under stress. The conductivity of acid-etched fractures in dolomite and limestone differs, primarily due to the varying deformation resistance of load-bearing protrusions under high closure stress as shown in Fig. 16. During testing, both dolomite and limestone samples exhibited progressive morphological deformation and localized fracture development within the acid-etched fractures. Morphological observations revealed that the etched zones remained structurally intact in both rock types. In contrast, changes occurred in the transition and under-etched zones, where the protrusions were primarily developed. These

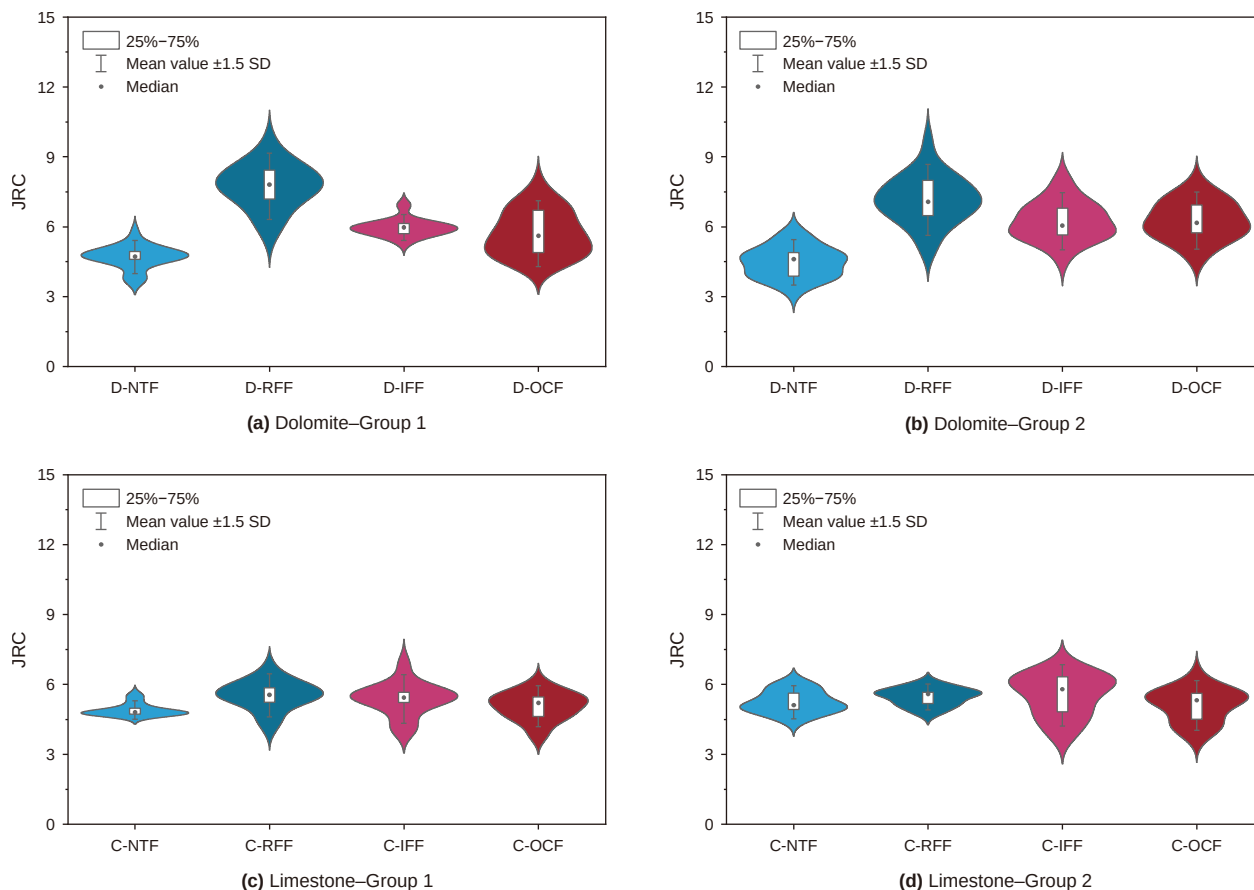


Fig. 11. JRC analysis of acid-etched fracture surfaces under different filled fracture conditions. (a, b) The acid-etched fracture surfaces of dolomite, (c, d) the acid-etched fracture surfaces of limestone.

protrusions exhibited distinct structural responses in different rock types: in dolomite, they induced secondary fractures around their margins; while in limestone, they tended to form indentations and embedding. In which, the influence of filled fractures is reflected in the spatial distribution between the etched and under-etched zones. The conductivity of dolomite exceeds that of limestone, indicating that under high closure stress, the load-bearing protrusion of fracture propagation mode is more effective in maintaining conductivity than the indentation-and-embedding deformation mode.

3.6. Sensitivity of interference modes to filled fracture width and injection rate

To examine the sensitivity of the observed interference behavior to key experimental parameters, additional acid-etching experiments were conducted on limestone samples by varying the width of the filled fracture and the injection rate under otherwise identical conditions, as shown in Fig. 17.

First, the injection rate was fixed at 30 mL/min, and the width of the filled fracture was increased from 2 to 3 and 4 mm. Under these conditions, the characteristic interference of both RFF and IFF remained unchanged, while the spatial extent of acid etching exhibited noticeable variations. For the RFF cases, the dual-track acid-etching pattern along the upper and lower boundaries of the main fracture surface was consistently observed. As the filled fracture width increased, the etched trajectories associated with the dual-track pattern became more pronounced and extended

farther downstream, indicating an enlargement of the local etched zones. For the IFF cases, the blocking effect of the inert filling became more evident with increasing fracture width. Acid etching remained concentrated in regions adjacent to the filled fracture, whereas the filled zone itself showed limited dissolution. In particular, areas located above the main fracture surface exhibited weaker etching compared to neighboring regions.

Second, the filled-fracture width of the RFF configuration was fixed at 2 mm, and the injection rate was varied from 30 to 50 and 10 mL/min. The preferential acid transport pathway characteristic of the RFF configuration was preserved across all tested injection rates, while the scale and intensity of etching varied. At the higher injection rate of 50 mL/min, acid etched zones developed along both the upper and lower boundaries of the main fracture surface, accompanied by an expanded under-etched region in the central part of the fracture. In this case, the dual-track etching pattern was apparent near the filled fracture, but became less distinct farther downstream. At the lower injection rate of 10 mL/min, the dual-track etching pattern was clearly delineated, manifested as deeper and more localized etch along the upper and lower boundaries of the main fracture surface. The relative depth of etching in these regions was significantly greater than that observed elsewhere along the fracture surface.

4. Discussion

The deep and ultra-deep carbonate reservoirs of the Tarim Basin record a complex tectono-diagenetic history that has

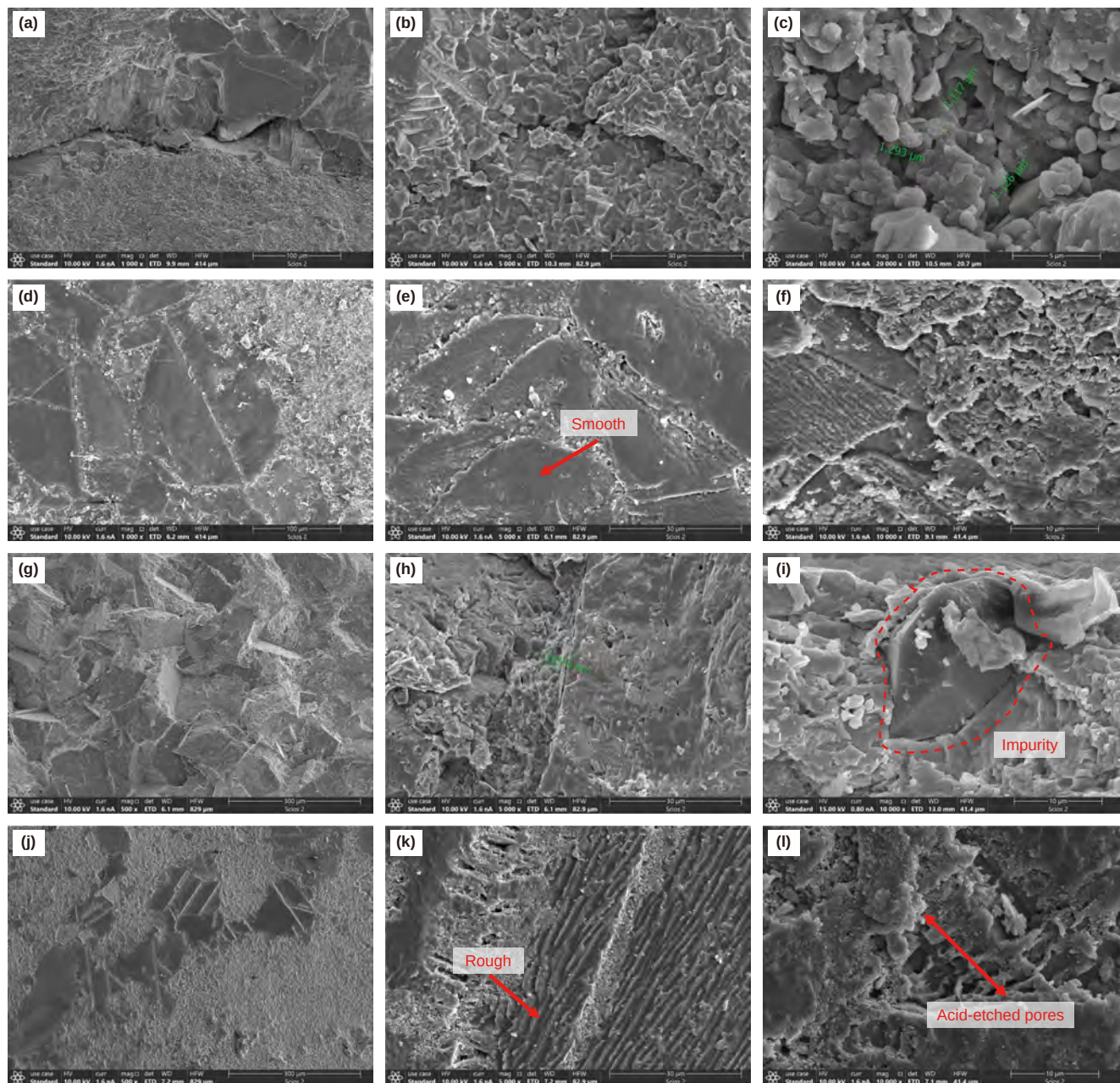


Fig. 12. SEM observations of fracture surfaces before and after acid etching in limestone and dolomite. (a–c) Micro-characteristics of limestone before acid-etching, (d–f) micro-characteristics of limestone after acid-etching, (g–i) micro-characteristics of dolomite before acid-etching, (j–l) micro-characteristics of dolomite after acid-etching.

produced a hierarchical system of fractures and dissolution cavities (Liu et al., 2022; Guan et al., 2019). Early tectonic activities generated high-angle fractures that later acted as conduits for diagenetic fluids and hydrothermal alterations (Zhang et al., 2011). With increasing burial depth, fracture fillings become progressively complex. The Yijianfang Formation primarily contains open or sparsely filled fractures, whereas the Wusongger and Xiaerbulake Formations commonly exhibit siliceous or partially filled fractures (Miller and Dunne, 1996; Duschl, 2018). Microscopically, the interlocking crystal textures and microfracture networks indicate multistage diagenetic modification (Bjørlykke and Høeg, 1997).

These stratigraphic differences provide the geological foundation for classifying the experimental interference modes. The OCF, RFF, and IFF used in this study correspond to the natural

occurrence of open, calcite-filled, and siliceous-filled fractures in deep carbonate formations. This correspondence enables a realistic representation of how filling states influence the evolution of acid-etched morphology and fracture conductivity during stimulation.

4.1. Interference mechanisms of filled fractures on acid transport

The interference of natural fractures with acid transport is fundamentally governed by the mineralogical composition and hydraulic properties of their fillings, which together determine local flow resistance and reaction kinetics. Variations in filling state alter the spatial coupling between acid transport and dissolution, producing three representative interference modes-

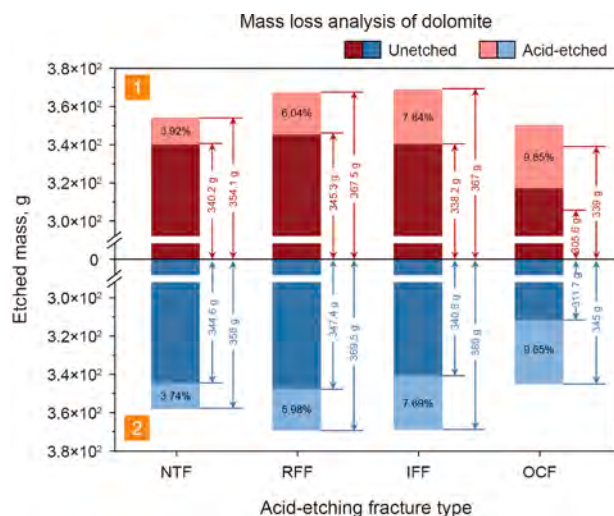


Fig. 13. Mass loss analysis of dolomite under different filled fracture conditions.

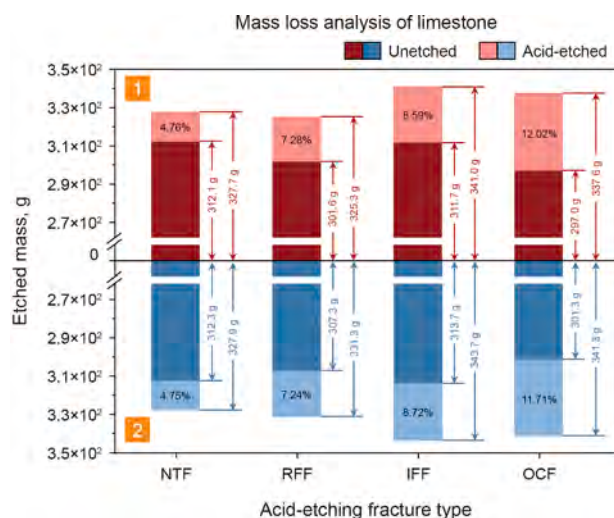


Fig. 14. Mass loss analysis of limestone under different filled fracture conditions.

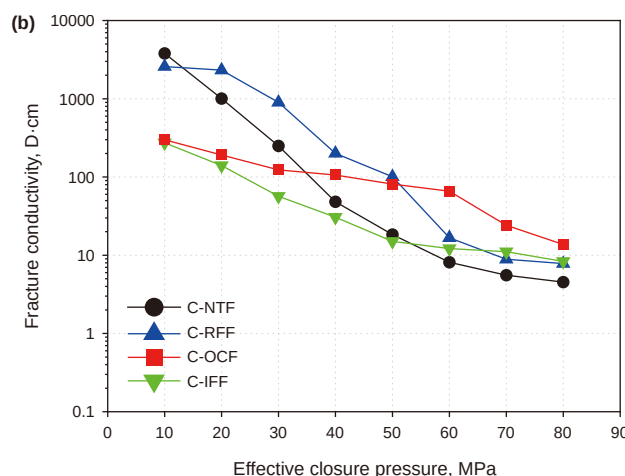
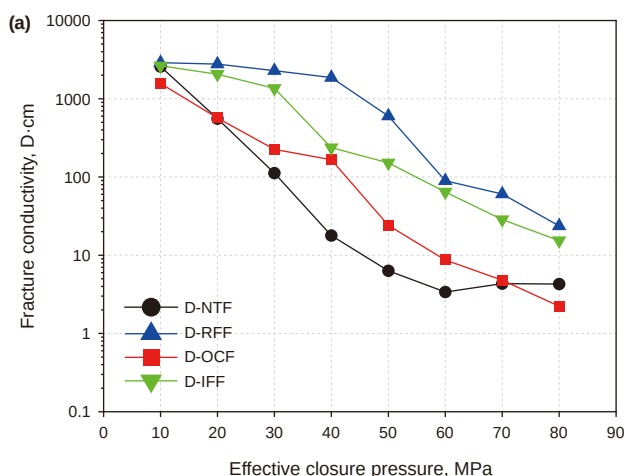


Fig. 15. Conductivity of acid-etched fracture. (a) Dolomite and (b) limestone.

deflection, obstruction, and bifurcation, which corresponding to RFF, IFF, and OCF, respectively (Fig. 18).

Fig. 19 presents the inlet pressure evolution during acid etching under different fracture-filling conditions for both dolomite and limestone samples. The inlet pressure responses exhibit distinct characteristics depending on the fracture-filling state. For dolomite, the OCF and IFF cases show generally smooth pressure curves with minor fluctuations characterized by low amplitude, narrow ranges, and long temporal intervals, resulting in a gently varying overall trend. Although the NTF case displays a larger number of fluctuation points, its overall pressure evolution remains comparable to that of the OCF and IFF cases. In contrast, the RFF case exhibits markedly different pressure behavior. The inlet pressure fluctuates more frequently and with larger amplitudes, forming a persistent zigzag pattern throughout the acid etching process, indicative of a highly unsteady pressure response. Similar trends are observed in the limestone experiments. In the OCF case, pressure fluctuations appear after approximately 1000 s but remain sparse and do not alter the overall smooth pressure trend. The IFF and NTF cases display comparable responses, with occasional fluctuations superimposed on relatively stable baselines. The RFF case again shows distinctive behavior, with relatively stable pressure at the early stage followed by pronounced and frequent fluctuations during the middle and late stages, characterized by wide pressure ranges and stepwise variations. The inlet pressure response provides indirect insight into changes in flow resistance during acid etching, reflecting the dynamic interaction between fluid transport and reaction-induced gas generation. For the OCF, IFF, and NTF cases, the pressure evolution does not show evidence of persistent flow blockage, as indicated by the generally stable pressure trends. In contrast, the RFF case is characterized by recurrent pressure buildup followed by rapid decay, a pattern that is consistent with intermittent flow interference associated with transient gas involvement during acid-filling interactions. Such pressure oscillations suggest that gas generated during the reaction may temporarily impede fluid flow but is progressively displaced or dissipated as injection continues, resulting in the observed oscillatory pressure response. It should be noted that, from a stoichiometric perspective, 1 mol of dolomite can theoretically release 2 mol of gas, whereas 1 mol of calcite releases only 1 mol (Eqs. (2) and (3)). However, under the experimental temperature of 150 °C, the dissolution rate of dolomite is lower than that of calcite, resulting in markedly different gas generation rates.

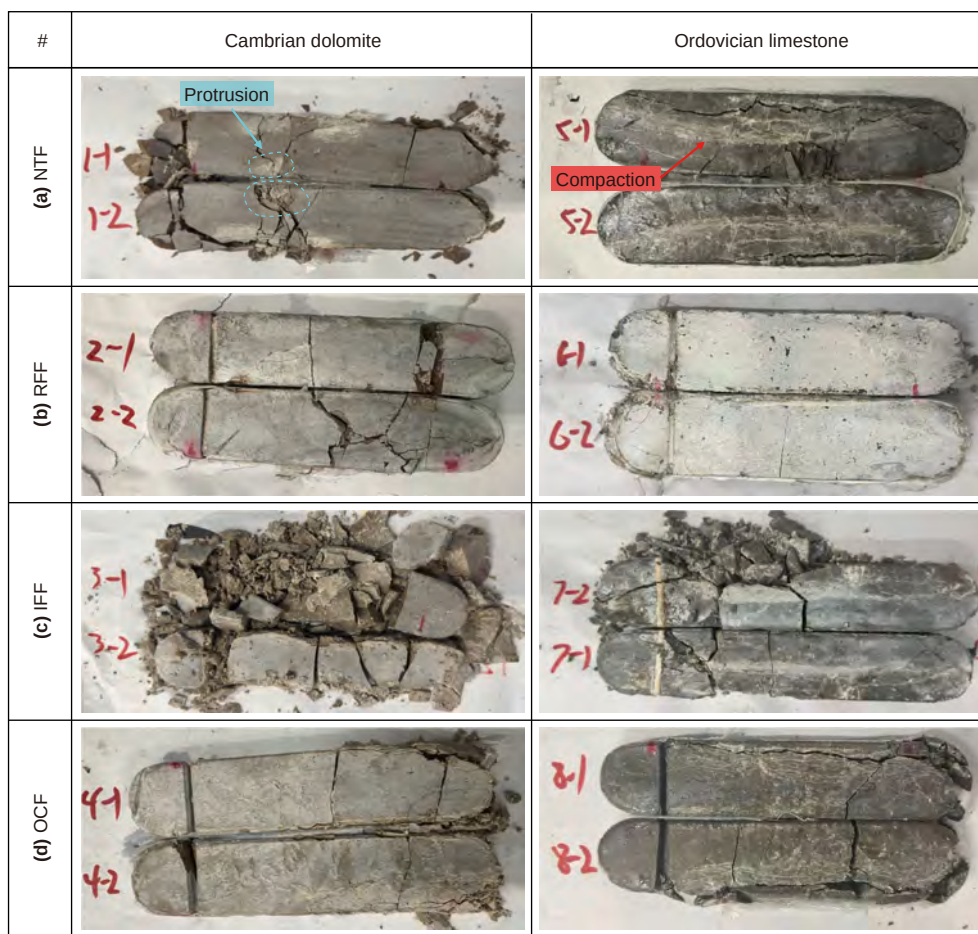


Fig. 16. Acid-etched fracture surface characteristics of the rock plate after the conductivity test under high closure stress.

Consequently, gas production is controlled not only by stoichiometry but also by reaction kinetics. For calcite, the relatively high dissolution rate promotes rapid gas release, which can lead to transient local gas accumulation and the formation of high-pressure zones near the reaction front. This short-term gas buildup alters the local flow field, causing the acid to divert toward both sides of the fracture rather than advancing uniformly, thereby forming a characteristic “double-track” dissolution pattern. In contrast, dolomite, despite its higher theoretical gas-generating potential, exhibits slower dissolution under high-temperature conditions. Gas is generated more gradually and is continuously transported out of the fracture with the flowing acid, reducing the intensity of gas-phase interference. These observations indicate that, in deep high-temperature carbonate formations, reaction kinetics rather than stoichiometric gas potential fundamentally govern gas–fluid interaction and its influence on acid transport behavior. Meanwhile, in the laboratory experiments, the outlet pressure of the fracture was maintained at 3 MPa, under which CO₂ exists in the gaseous state, driving flow deflection through significant volumetric expansion. In contrast, under ultra-deep field conditions, the bottom-hole pressure far exceeds the critical pressure (7.38 MPa), causing CO₂ to transition into a supercritical state. Although the volumetric expansion of SC-CO₂ is limited compared to the gas phase, flow hindrance under these conditions is governed primarily by multiphase immiscibility and capillary forces. The high interfacial tension between the aqueous acid and the non-wetting supercritical CO₂ phase generates substantial capillary resistance within narrow flow channels. Combined with

the distinct viscosity contrast, these mechanisms sustain strong flow interference during field-scale acidizing, validating the applicability of the proposed deflection model to ultra-deep reservoir conditions (see Fig. 20).

Based on the combined observations of etching morphology, penetration depth, and inlet pressure dynamics, the acid–fracture interaction under different fracture-filling conditions can be interpreted in terms of three characteristic flow-interference modes. In the deflection mode (RFF), acid–calcite reactions induce transient pressure buildup within the filled zones. The increased local resistance redirects the acid toward adjacent low-resistance channels along the fracture margins, resulting in a dual-channel dissolution pattern. This configuration enhances penetration depth while maintaining moderate overall acid consumption, reflecting a balanced chemo-hydraulic control on flow partitioning. The obstruction mode (IFF) occurs when siliceous fillings impede both reaction and flow. Acid accumulation upstream raises the local pressure gradient until the flow is redistributed along the filling boundaries. The resulting sheet-like etching belts are laterally confined and shallow, indicating limited transport efficiency and reduced penetration capability. In the bifurcation mode (OCF), open fractures provide high-conductivity leak-off pathways. Acid preferentially invades these conduits, producing intense etching at the intersection but rapid depletion downstream. The bifurcated flow leads to strong near-well dissolution yet poor deep propagation. It should be noted that, the mass loss of OCF is greater than that of RFF. This mass loss disparity verifies our hypothesis regarding deep acid penetration. The RFF

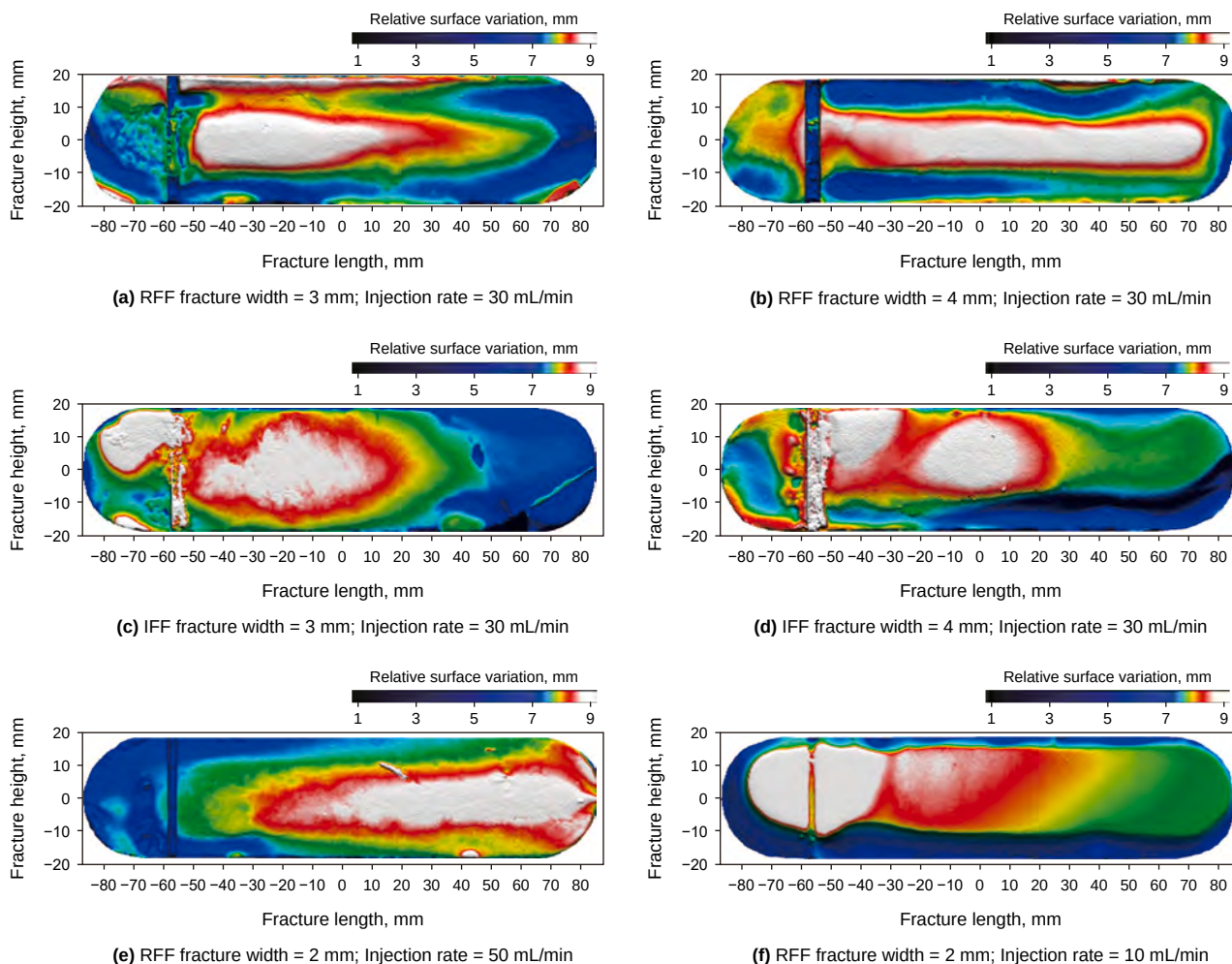


Fig. 17. Effects of filled fracture width and injection rate on acid-etching morphology in limestone fractures.

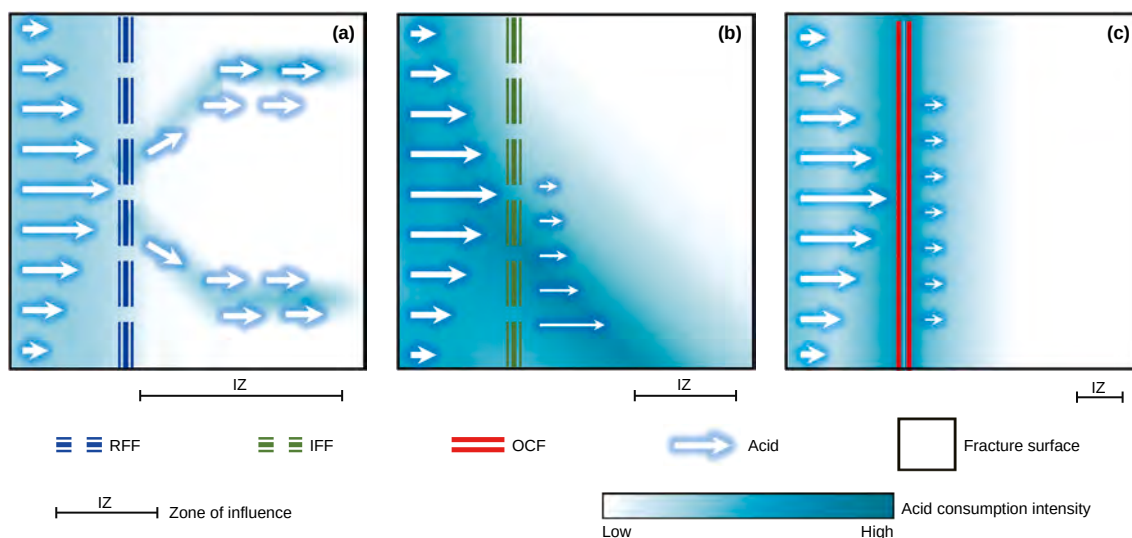


Fig. 18. Schematic diagram of three interference modes of filled fractures on acid fluid transport.

scenario induces a ‘confined channeling’ flow regime (track-shaped etch). By disturbance acid flow into narrow, high-velocity grooves, the contact area-to-volume ratio is reduced, effectively

creating an acid retardation effect. This preserves the acid strength for deeper penetration. Conversely, the OCF scenario is characterized by ‘distributed dissolution’ (acid leak-off + sheet-like etch).

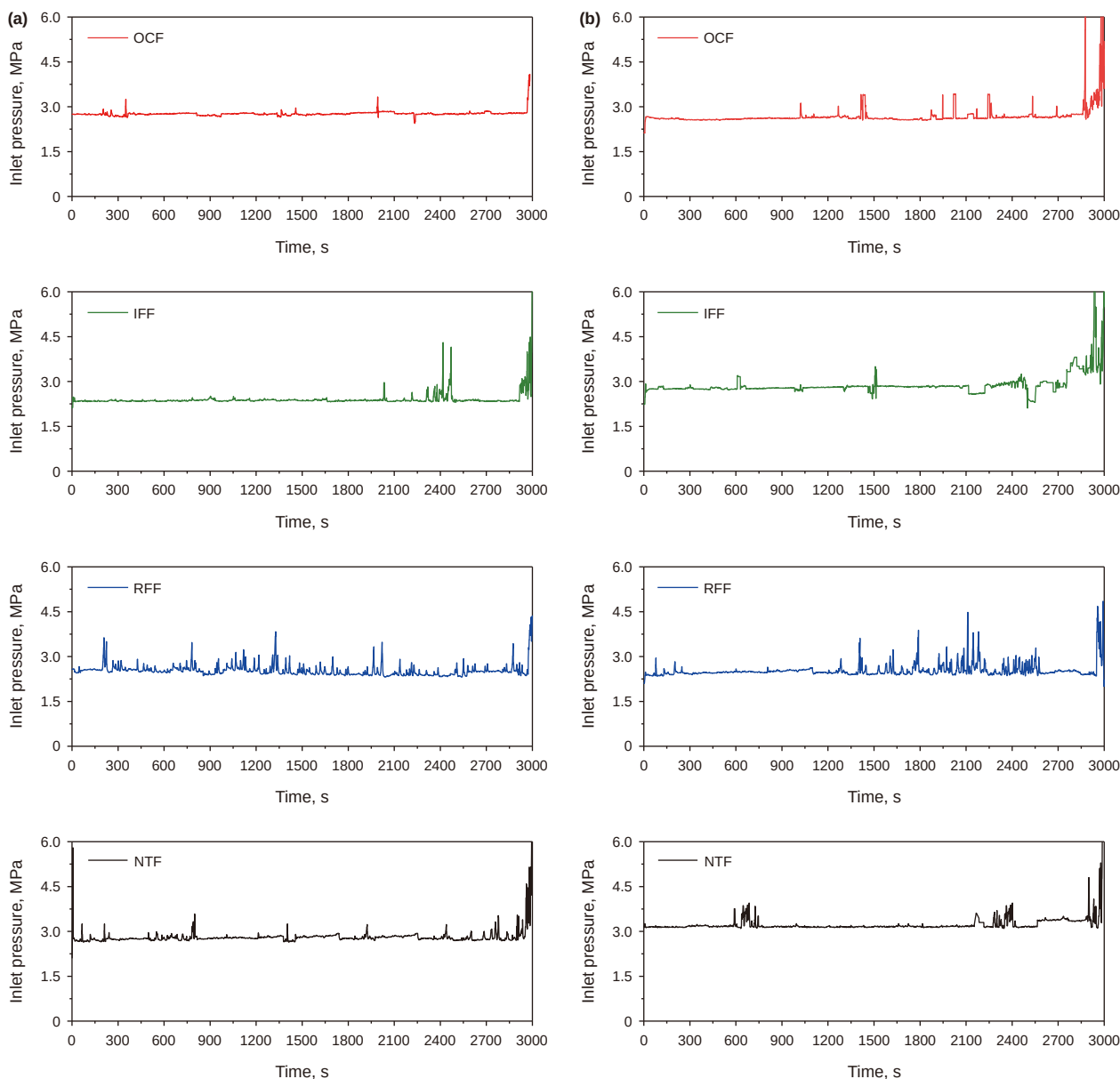


Fig. 19. Inlet pressure response during acid etching under different fracture-filling conditions. (a) Dolomite and (b) limestone.

The bifurcated flow exposes the acid to a vast reactive surface area simultaneously. While this results in high mass loss, it leads to rapid near-wellbore acid depletion, severely compromising the capacity for deep reservoir stimulation.

Collectively, these observations establish a coupled chemo-hydraulic framework that links fracture-filling state to acid: reactive fillings induce deflected channelization, inert fillings cause confined obstruction, and open conduits lead to premature leak-off. Sensitivity analyses confirm that while injection parameters affect the etching scale, the fundamental interference patterns remain stable. Regarding fracture continuity, the ‘fully filled’ and ‘open’ configurations investigated here represent hydraulic end-members; thus, natural partially filled fractures can be interpreted as intermediate states bounded by these extremes. Regarding the physical representation of fracture fillings, it is acknowledged that the compacted particulate materials used in the experiments possess higher intrinsic porosity than natural mineral veins. However, the effective hydraulic obstruction

observed in the IFF case is primarily attributed to the rheological properties of the acid system rather than the tightness of the filling material itself. The cross-linked acid system, designed for ultra-deep reservoirs, exhibits high viscosity and strong wall-building capabilities. These properties create a viscous seal or filter cake at the interface of the porous filling, effectively inhibiting permeation into the matrix. Combined with the short residence time (~4.5 s) in the fracture, this ensures the system operates in an advection-dominated regime. Consequently, acid transport is governed by fracture-scale flow partitioning around the fillings rather than Darcy flow permeation through them, thereby reproducing the macroscopic interference behavior expected in tight natural fractures. The influence of reservoir temperature on these interference mechanisms is twofold, affecting both reaction kinetics and fluid rheology. At lower temperatures (~120 °C), slower reactions favor more uniform acid spending, resulting in weaker chemical-hydraulic coupling. At intermediate temperatures (~150 °C), representative of many ultra-deep carbonate reservoirs

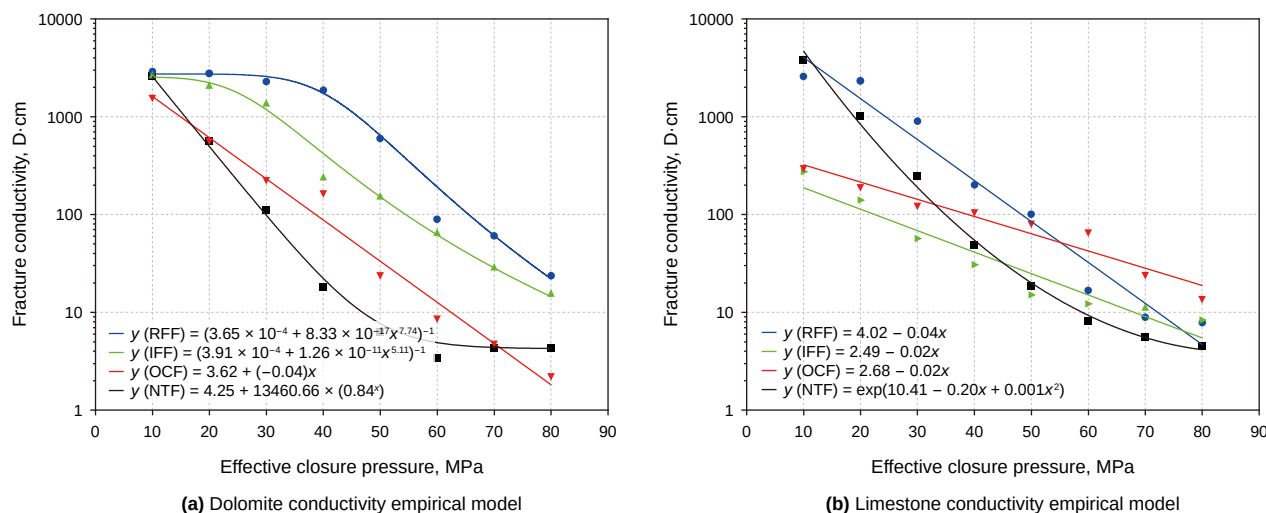


Fig. 20. A fitting empirical model for the conductivity of acid-etched fractures with filled fracture. (a) Dolomite and (b) limestone.

in the Tarim Basin, reaction rates are sufficient to amplify the contrast between reactive- and inert-filled fractures, while the cross-linked acid retains its viscosity and structural integrity. This regime supports the stable interference modes (deflection, obstruction) identified in this study. However, at ultra-high temperatures ($\geq 180^\circ\text{C}$), the system enters a critical transition. Beyond accelerating reaction rates to a transport-limited regime, thermal degradation of the polymer network may cause a sharp reduction in acid viscosity. In this scenario, the flow regime could shift from being controlled by fracture-filling structures to being dominated by viscous fingering and random dissolution instabilities. Therefore, the systematic interference modes proposed here are strictly valid within the thermal stability window of the acid system. Consequently, within this stability range, the observed interference behavior is controlled mainly by the presence and reactivity of the fillings, rather than by their internal permeability. Although the laboratory specimens ($178\text{ mm} \times 38\text{ mm} \times 20\text{ mm}$) are shorter than natural fractures, the governing reaction-transport processes and the characteristic etched and transition zone distributions identified in this study are expected to remain representative at larger scales, provided that the hierarchy of hydraulic conductance is preserved.

4.2. Coupled influence of etched morphology and rock mechanics on fracture conductivity

The identified interference modes dictate the acid-etched surface topography, which fundamentally governs residual fracture conductivity. However, the physical mechanism governing conductivity loss under stress is the evolution of the fracture contact area. Sustaining this conductivity under closure stress relies on preserving the hydraulic aperture of the differentially etched channels. Consequently, filled fractures influence conductivity by modulating the geometry and continuity of these flow paths. Although the absolute conductivity values are low at high stress, the relative differences among different fracture-filling conditions remain systematic and reproducible. It is crucial to explicitly distinguish between the deformation mechanisms captured in the laboratory versus those operative in the field. The conductivity measurements, conducted with a standard 30-min hold time, primarily reflect the instantaneous plastic embedment and elastic deformation of asperities under effective stress. They do not fully account for time-dependent creep, a slow viscoplastic

deformation process that becomes critical over the multi-year lifespan of a well. Carbonate rocks, particularly limestone, are prone to significant creep deformation under high stress (Li et al., 2018a, 2018b). This ductile behavior leads to a rapid expansion of the contact area as asperities flatten, effectively choking the flow channels. Therefore, the reported conductivity for limestone likely represents an upper bound, with further degradation expected over time. In contrast, the higher creep resistance and brittleness of dolomite allow asperities to fail by brittle fracture rather than viscoplastic flow, thereby limiting contact area growth. This suggests that the observed superiority of dolomite over limestone would be even more accentuated in long-term production. Ultimately, these findings indicate that while acid etching modifies the initial morphology, the long-term stability of the fracture is intrinsically linked to the rock's mechanical ability to resist contact area expansion via creep.

Across all cases, dolomite exhibited higher conductivity than limestone, reflecting its stronger mechanical framework and more anisotropic acid-etched morphology. In dolomite, the deflection mode (RFF) produced deep, connected channels that maintained high conductivity at low closure stress, whereas increasing stress progressively narrowed these channels, reducing the lithologic contrast once compression dominated. In contrast, limestone displayed a uniform, linear decline in conductivity with increasing stress for all interference modes; smoother surfaces and weaker frameworks promoted plastic compaction and early closure. Dolomite retained greater variability and higher residual conductivity, confirming that anisotropic etched morphology contributes more effectively to maintaining conductive pathways under stress. To quantitatively evaluate the influence of different fracture-filling states on the conductivity of acid-etched fractures, an empirical conductivity prediction model was developed to describe the relationship between fracture conductivity and closure stress for two lithologies. The model was established by fitting the experimentally measured conductivity data of acid-etched fractures with different filling conditions as a function of closure stress. The results indicate that acid-etched fractures in dolomite exhibit a nonlinear, exponential decay in conductivity with increasing closure stress. Under the RFF and IFF conditions, fractures show relatively strong resistance to deformation at the early stage, whereas conductivity degradation in the OCF and NTF cases occurs more rapidly. In contrast, acid-etched fractures in limestone display an approximately linear decline in conductivity and

generally exhibit poor resistance to closure stress. According to the fitted model, fracture fillings primarily influence the early-stage conductivity of acid-etched fractures, which may lead to high initial productivity in field operations. However, as closure stress accumulates over time, fracture closure accelerates and conductivity decays significantly, resulting in a decline in production performance. It should be noted that the proposed model is empirical in nature and is applicable within the experimental conditions and geometrically similar parameter ranges investigated in this study.

Although calcite exhibits faster intrinsic reaction kinetics with acid compared to dolomite, this rapid reactivity does not necessarily translate into higher fracture-scale roughness. SEM observations reveal that acid etching in limestone is dominated by relatively uniform surface retreat at the crystal scale. This uniform dissolution reduces the height contrast of asperities, thereby promoting smoother contact surfaces that are prone to closure. In contrast, the slower and more selective dissolution of dolomite facilitates heterogeneous etching along crystal boundaries and defects, generating a complex, multiscale roughness that is structurally more favorable for sustaining fracture conductivity. It is crucial to interpret these morphological differences within the context of the reaction regime. Under the high-temperature conditions (150 °C) employed in this study, acid-carbonate reactions are likely dominated by mass transport rather than intrinsic surface kinetics. In this transport-limited regime, the instantaneous dissolution rate is governed by flow dynamics rather than the stoichiometric acid demand per mole of mineral. While mineral stoichiometry determines the ultimate dissolution capacity—providing a secondary context for total mass loss—it is the fracture geometry and flow partitioning that primarily dictate the interference mechanisms. However, a favorable etching morphology alone cannot guarantee long-term conductivity; the mechanical integrity of these etched asperities under stress is equally critical. To quantify the acid-induced mechanical degradation and its impact on fracture stability, mechanical parameters were measured for both lithologies before and after acid etching (Table 3). The results confirm significant but distinct patterns of degradation. For dolomite, a sharp decline in both compressive strength (from 399.16 to 53.34 MPa) and tensile strength (from 19.19 to 8.57 MPa) was observed. This dramatic reduction indicates a shift towards a structure prone to brittle failure, which paradoxically aids in forming supporting rubble or asperities. Conversely, the primary alteration in limestone was a substantial drop in elastic modulus (from 59.24 to 12.21 GPa). This significant loss of stiffness implies that the limestone matrix becomes highly compliant and deformable post-etching, leading to asperity embedment and conductivity loss under high closure stress.

At the same time, rock mechanics tests for different formation (Fig. 21) supports these results: the Wusongger dolomite shows higher compressive strength and elastic modulus than the Yijianfang limestone, consistent with its brittle deformation and ability to preserve channel openness. Limestone, characterized by lower strength and ductile behavior, undergoes pronounced compaction, while Xiaerbulake samples exhibit low and scattered strength due to microfracture development. Despite the low absolute conductivity values at high stress, the conductivity responses of different fracture-filling conditions remain distinguishable within the same stress range. The observed trends indicate strong stress sensitivity of acid-etched fracture surfaces in both lithologies under high closure stress.

Overall, the results demonstrate a coupled morphological-mechanical control mechanism on fracture conductivity. The acid-filling interaction dictates the etched geometry, whereas the mechanical competence of the rock determines its ability to

Table 3

Comparison of rock mechanical parameters for pre- and post-etched carbonate samples.

Sample	Status	Tensile strength, MPa	Compressive strength, MPa	Poisson's ratio	Elastic modulus, GPa
# Dolomite	Pre-etching	19.19	399.16	0.248	67.44
	Post-etching	8.57	53.339	0.076	43.58
# Limestone	Pre-etching	9.13	183.09	0.226	59.24
	Post-etching	8.70	74.784	0.11	12.21

retain conductive space under stress. Additionally, the potential for fines migration from insoluble residues—particularly in impurity-rich dolomite—presents a secondary risk for conductivity impairment. However, our data suggest that under high closure stress, the positive impact of dolomite's mechanical strength outweighs the negative effects of residue plugging. This coupling highlights that the effectiveness of acid fracturing in deep carbonates depends on coordinating acid-etch pattern formation with the stress sensitivity of different lithologies. The low fracture conductivity observed under high closure stress reflects the intrinsic mechanical behavior of deep carbonate rocks rather than insufficient acid etching. Under ultra-deep reservoir conditions, fracture surfaces are subjected to severe closure stress (125–180 MPa), leading to rapid asperity crushing and plastic deformation, particularly in limestone. Therefore, low absolute conductivity values at high stress are expected and consistent with field observations.

4.3. Scaling considerations and dimensionless analysis

A pronounced scale disparity exists between laboratory experiments and field applications, particularly in hydraulic parameters (e.g., injection rate and acid volume) and fracture geometry, with field operations occurring at scales several orders of magnitude larger. To address this discrepancy, a dimensionless analysis is introduced to characterize acid transport behavior across scales. Specifically, the Péclet number (Pe), which quantifies the balance between advective and diffusive transport, is adopted as the primary scaling criterion and is defined in Eq. (4). The Péclet number is adopted for scale conversion because acid transport within fractures is predominantly governed by laminar flow. In laboratory experiments, the injection rates are sufficiently low to ensure laminar conditions. Although field-scale operations involve larger fracture dimensions and higher injection rates, the flow velocity decreases and stabilizes as the acid penetrates deeper into the fracture network. Under these conditions, the transport regime transitions back to laminar flow, making the Péclet number an appropriate scaling parameter for characterizing acid transport across laboratory and field scales.

$$Pe = \frac{ub}{D} \quad (4)$$

where Pe is the Péclet number; u is the mean flow velocity in the fracture, $\text{m}\cdot\text{s}^{-1}$; b is the fracture aperture, m ; and D is molecular diffusivity, $\text{m}^2\cdot\text{s}^{-1}$.

Based on the experimental geometry (30 mL/min through a 2 mm fracture gap), the characteristic mean velocity is estimated as Eq. (5):

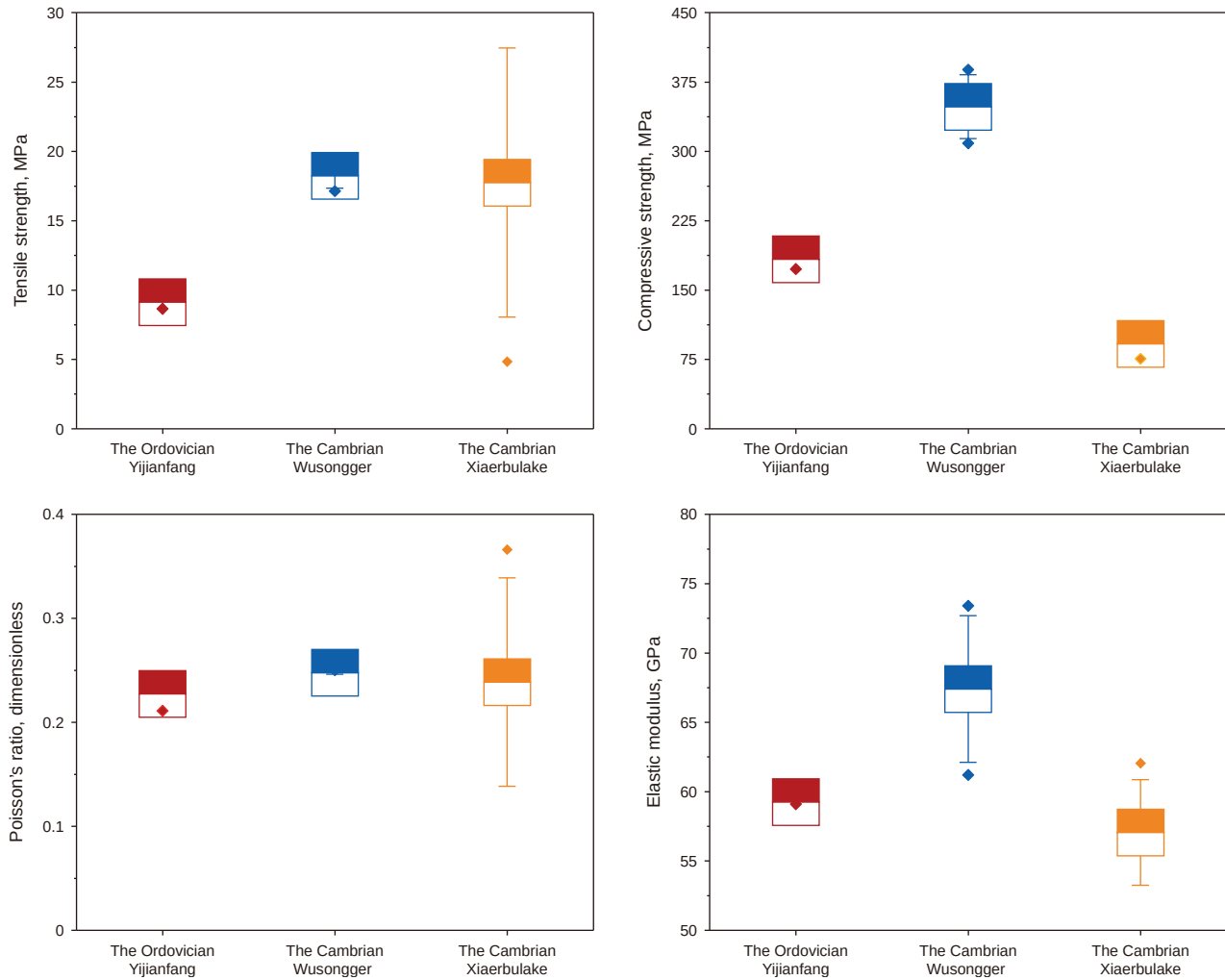


Fig. 21. Rock mechanics test results of dolomite and limestone.

$$u = \frac{Q}{bh} \quad (5)$$

where Q is the injection flow rate, $\text{m}^3 \cdot \text{s}^{-1}$; and h is the fracture height, m.

Under laboratory conditions, a smooth rock slab is used to represent the initial fracture surface, resulting in a relatively simple acid-flow pattern at the early stage. At the applied low injection rates, the flow regime remains laminar. The filled fracture is positioned 30 mm downstream from the acid inlet. Based on Eq. (5), the estimated transport time for the acid to reach the filled fracture is approximately 4.5 s. As demonstrated in Section 3.2, temperature is the primary factor controlling acid viscosity. Under the experimental temperature of 150 °C, the viscosity of the acid solution remains relatively stable before and after acid etching, within a range of approximately 60–90 mPa·s. These values are subsequently used in the Reynolds number calculation to evaluate the flow regime during the experiments.

$$Re = \frac{\rho ub}{\mu} \quad (6)$$

where Re is the Reynolds number; ρ is the fluid density, $\text{kg} \cdot \text{m}^{-3}$; and μ is the dynamic viscosity, Pa·s.

Substituting these values into the Reynolds number formulation yields Reynolds numbers on the order of 0.15–0.22 (assuming

$\rho \approx 1000 \text{ kg} \cdot \text{m}^{-3}$), confirming that the fracture flow remains within the laminar regime. The corresponding Péclet numbers are estimated to be on the order of 10^3 – 10^4 , based on typical molecular diffusivities of $D = 10^{-8}$ – $10^{-9} \text{ m}^2 \cdot \text{s}^{-1}$, indicating that acid transport within the fracture is predominantly advection-dominated. These transport characteristics are consistent with flow conditions commonly encountered in deep carbonate fracture systems, where laminar flow and convection-controlled transport prevail.

Although field-scale fractures are larger and hydraulic parameters are more variable, the three interference patterns identified in the laboratory remain applicable. As demonstrated by the laboratory results (Section 3.6), these patterns are primarily controlled by the filling state and geometric configuration of intersecting fractures, whereas fracture scale and hydraulic parameters mainly influence the extent and depth of etching rather than the interference mode itself. To provide a standardized evaluation criterion, under laminar flow conditions, the relative hydraulic conductance of competing flow paths can be used to distinguish the three interference patterns associated with different fracture-filling types.

$$G \propto \frac{b^3}{\mu L} \quad (7)$$

where G is the hydraulic conductance of fracture and L is the characteristic flow length, m.

As long as the hierarchy of hydraulic conductance imposed by fracture filling configurations is preserved, the qualitative interference patterns are expected to remain valid across scales. Therefore, changes in scale are expected to mainly affect the magnitude of penetration and residence time (i.e., the spatial extent of etched, transition, and under-etched zones), whereas the interference patterns associated with different filling states remain qualitatively consistent. Furthermore, it is essential to consider the thermal dynamics in field operations. Unlike the isothermal conditions (150 °C) maintained in the laboratory, field acidizing involves a significant temperature gradient. The injection of cool fluids retards the reaction rate in the near-wellbore region, preserving acid strength for deeper penetration. Consequently, the laboratory results, obtained under immediate exposure to maximum reservoir temperature, represent a conservative scenario, suggesting that field-scale penetration would likely exceed laboratory predictions. A full field-scale translation requires matching the relevant dimensionless groups and incorporating multiphase effects and field fracture geometries.

To further quantify the validity of the reaction regime, the Damköhler number (Da), representing the ratio of the reaction rate to the convective transport rate, is introduced:

$$Da = \frac{kL}{ub} \quad (8)$$

where Da is the Damköhler number and k is the reaction rate constant.

Based on the kinetics data from Lund et al. (1975), the reaction rate constant k for acid-calcite at 150 °C is estimated to be in the order of 10^{-4} to 10^{-3} m/s. With a mean flow velocity $u \approx 0.0066$ m/s and $L = 0.178$ m, the calculated Damköhler number ranges from 2.7 to 27 ($Da \gg 1$). These observations indicate that acid consumption occurs rapidly relative to the fluid flow rate. As a result, even with the limited specimen dimensions used in the laboratory, measurable acid consumption and associated changes in etching morphology can still develop. This demonstrates that the experimental system successfully captures the essential characteristics of acid-rock interaction. Consequently, similar acid-etching and transport behaviors are expected to persist during fracture acidizing at the field scale.

Finally, to quantitatively compare the stimulation performance of different fracture-filling scenarios, a dimensionless stimulation efficiency index (ASEI) is introduced. The index is defined as the ratio of normalized effective penetration length (etched zones) to normalized acid consumption, i.e.,

$$ASEI = \frac{L_{\text{eff}}/L_0}{\Delta m/m_0} \quad (9)$$

where ASEI is the dimensionless stimulation efficiency index; L_{eff} is the effective acid-etched penetration length; L_0 is the initial length of the rock sample or fracture; Δm is the acid induced mass loss and m_0 is the initial mass of the rock sample.

A higher ASEI value indicates more effective penetration per unit acid consumption and thus higher stimulation efficiency. ASEI values show clear differences among the four scenarios: the RFF configuration exhibits the highest ASEI, reflecting a favorable balance between penetration depth and acid consumption; the IFF case shows a moderate ASEI, indicating constrained penetration with intermediate acid utilization efficiency. In contrast, the OCF case yields a low ASEI despite large mass loss, due to excessive acid leakoff and limited effective penetration. The NTF case exhibits a moderate stimulation efficiency, reflecting a relatively uniform acid etching behavior in the absence of fracture-filling

interference, but without the formation of preferential acid transport pathways. This dimensionless evaluation provides a unified framework to compare stimulation efficiency across different interference modes and highlights the cost-effectiveness of the RFF scenario.

The meanings of symbols are shown in the Nomenclature below.

4.4. Geological and engineering implications

In ultra-deep reservoirs, drilling and completion costs are high and construction periods are long, making it essential to maximize production per well. Hydrocarbon resources in the Tarim Basin are typically characterized by dispersed distribution, trap-controlled accumulation, and ultra-low matrix permeability. Under such conditions, the effectiveness of each artificial fracture becomes critical and is jointly influenced by natural fracture distribution, acid penetration behavior, and the evolution of fracture conductivity.

For dispersed fracture-cavity carbonate reservoirs (e.g., the Tahe Oilfield), stimulation design should focus on improving the directional connectivity and effective reach of artificial fractures. A staged treatment strategy is recommended, in which high-viscosity, high-rate fracturing fluids are first used to generate artificial fractures and establish connectivity with isolated or remote fracture-cavity bodies. This is followed by closure acidizing to enhance long-term fracture conductivity. Interaction with natural fractures is inevitable in such reservoirs; therefore, active management of fracture intersections, rather than complete avoidance, is required. In particular, intersections with OCF may lead to excessive acid leak-off, while IFF can locally restrict forward acid transport. These effects should be mitigated through appropriate fluid design, injection sequencing, and diversion strategies to preserve effective acid penetration along the target fracture. For fault-controlled, concentrated carbonate reservoirs (e.g., the Shunbei Oilfield), stimulation design should emphasize activation and utilization of fault-related fracture systems (Yang et al., 2022). Hydrocarbons in these reservoirs are commonly distributed along strike-slip fault zones, where OCF are well developed and highly connected. Artificial fractures should be guided to intersect and activate these fault zones to establish high-conductivity flow paths. Once effective communication between artificial fractures and fault-related natural fractures is achieved, large-volume acid injection can be applied to extensively modify both natural and artificial fracture surfaces. In this context, controlled interaction with OCF is beneficial, while the influence of IFF should be carefully managed to avoid localized obstruction of acid transport.

Overall, interaction with natural fractures should be regarded as an inherent component of acid fracturing in ultra-deep carbonate reservoirs. Engineering design should therefore focus on managing flow partitioning and interference effects induced by different fracture-filling states, rather than attempting to avoid fracture intersections. Based on the identified interference mechanisms, a matching relationship between fracture-filling state and acid-fracturing operational parameters can be established. RFF promote deflected but continuous channelization; thus, moderate-to-high injection rates combined with medium-to-high acid viscosity are favorable for sustaining penetration while limiting excessive near-well acid spending. IFF impose strong flow obstruction, requiring higher displacement and larger acid volumes to overcome local resistance, whereas excessively high viscosity may further aggravate blockage. In contrast, OCF facilitate rapid leak-off, for which lower-viscosity fluids, controlled injection rates, and staged acid volumes are more effective. The NTF

Table 4

Quantitative design ranges for acidizing in Tahe and Shunbei carbonate reservoirs (field-based guidelines).

Parameter	Tahe Oilfield	Shunbei Oilfield	Engineering rationale
Reservoir depth	5400–6900 m	>7000 m	Controls pressure, temperature, and acid stability
Temperature	120–150 °C	160–200 °C	Determines acid type and reaction control
Dominant reservoir type	Fracture–vug carbonate	Fault–karst carbonate	Governs interference modes
Acid system	Gelled/cross-linked acid (20% HCl)	Surface cross-linked acid + authigenic acid	Field-proven systems
Pumping rate	5.5–6.6 m ³ /min	0.5–14 m ³ /min (stage-dependent)	High rate extends acid penetration
Single-stage acid volume	200–350 m ³	200–1000 m ³	Matched to cavity scale and leakoff risk
Preflush volume	10%–25% of total acid	10%–20% of total acid	Reduce near-well damage
Diversion strategy	Conditional (fracture zones)	Frequent multi-cycle diversion	Control dominant channels
Effective diversion criterion	ΔP increase $\geq$ 3–8 MPa	ΔP increase $\geq$ 5–12 MPa	Pressure response indicator X

represents a baseline condition where conventional acid-fracturing parameters are generally applicable.

These mechanistic insights are consistent with field practices in the Tarim Basin (Wang et al., 2016). In the Tahe Oilfield, acidizing primarily targets near-wellbore damage removal and fracture-cavity connectivity, and operational parameters remain relatively stable, with pumping rates typically ranging from 5.5 to 6.6 m³/min and single-stage acid volumes of 200–350 m³ using gelled or cross-linked acid systems. In contrast, the Shunbei Oilfield is characterized by ultra-deep, ultra-high-pressure fault–karst reservoirs, where acidizing aims at deep penetration and activation of large-scale natural fractures (Jiang et al., 2019; Li et al., 2021). Consequently, pumping rates and acid volumes span much wider ranges (0.5–14 m³/min and 200–1000 m³ per stage), with multi-stage acid systems and diversion cycles commonly applied. These quantitative ranges provide practical constraints for translating the identified interference modes into field-scale acidizing design, as summarized in Table 4. To translate these interference modes into field-scale numerical simulations (e.g., using FracPro or Kinetix), it is recommended to adjust the leak-off coefficient (C_L) and reaction parameters based on the identified fracture-filling characteristics: ① The bifurcation mode implies severe fluid loss. It is suggested to apply a high C_L multiplier (e.g., >2.0) to capture the rapid depletion of acid into the open network; ② The deflection mode maintains main fracture propagation but increases local consumption. A moderate C_L combined with an enhanced reaction rate factor is appropriate to simulate the dual-track dissolution; ③ The obstruction mode acts as a flow barrier. A reduced C_L or a transmissibility multiplier <1.0 should be applied to model the hindrance to acid penetration.

In summary, for both dispersed and fault-controlled ultra-deep formations, production enhancement depends on precise, targeted control of artificial fracture geometry and propagation direction. This targeted artificial fracture not only establishes formation connectivity but also prevents acid deflection and leakage, ensuring deep penetration, which will be the primary focus of subsequent work.

5. Conclusions

This study establishes a geology-engineering integrated framework to elucidate how natural fracture filling states govern acid transport and etched-fracture conductivity in ultra-deep carbonate formations of the Tarim Basin. The following conclusions can be drawn:

- (1) Natural fracture heterogeneity: Ultra-deep carbonate formations exhibit high-angle, multi-scale fracture systems with diverse mineral fillings. Ordovician formations mainly contain open or partially calcite-filled fractures, whereas Cambrian formations are dominated by silica-filled or incompletely filled cavities, reflecting distinct diagenetic evolution.

- (2) Fracture filling-controlled acid interference: The interaction between acid and fractures is primarily governed by the mineralogical nature of the fillings. Open-conduit fractures (OCF) act as physical bifurcation channels that promote severe acid leakage, inert-filled fractures (IFF) composed of siliceous minerals behave as impermeable barriers restricting acid penetration, and reactive-filled fractures (RFF) composed of calcite induce coupled chemo-physical flow diversion through localized dissolution and gas accumulation. These distinct interference mechanisms lead to significant variations in acid etching morphology and utilization efficiency.
- (3) Acid transport and etching efficiency: In dolomite, mass loss follows OCF > IFF > RFF, while acid penetration and conductivity exhibit the opposite trend (RFF > IFF > OCF), revealing that excessive leak-off lowers acid utilization. Limestone undergoes stronger overall dissolution but produces smoother surfaces with lower roughness and conductivity, indicating inefficient etching.
- (4) Conductivity and mechanical response: Under high closure stress, dolomite maintains higher conductivity than limestone due to its brittle failure and stronger resistance to deformation. In contrast, limestone experiences plastic compaction and rapid conductivity decay, confirming the superior stability of dolomite acid-etched fractures in deep reservoirs.

CRediT authorship contribution statement

Yao-Yao Sun: Writing – original draft, Investigation, Data curation. **Shuai Wang:** Validation, Data curation. **Xiao-Guang Wu:** Funding acquisition, Conceptualization. **Gen-Sheng Li:** Writing – review & editing, Supervision, Funding acquisition. **Zhong-Wei Huang:** Writing – review & editing, Supervision, Funding acquisition. **Jian-Ye Mou:** Methodology. **Zi-Xiao Xie:** Writing – review & editing, Visualization. **Wen-Chao Zou:** Software.

Data availability

Data will be made available on request.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.petsci.2026.05.009>.

Nomenclature

Q	Injection flow rate, $\text{m}^3 \cdot \text{s}^{-1}$
b	Fracture aperture, m
h	Fracture height, m
A	Effective flow cross-sectional area, m^2
u	Mean flow velocity in the fracture, $\text{m} \cdot \text{s}^{-1}$
L	Characteristic flow length, m
t	Residence time, s
μ	Dynamic viscosity, $\text{Pa} \cdot \text{s}$
ρ	Fluid density, $\text{kg} \cdot \text{m}^{-3}$
D	Molecular diffusivity, $\text{m}^2 \cdot \text{s}^{-1}$
k	The reaction rate constant
Da	The Damköhler number
Re	Reynolds number
Pe	Péclet number
G	Hydraulic conductance of fracture
$ASEI$	Dimensionless stimulation efficiency index
L_{eff}	The effective acid-etched penetration length
Δm	The acid-induced mass loss
C_L	The leak-off coefficient

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