



Research article

Interfacial fracture toughness of barite rocks

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ABSTRACT

This paper presents the results of an investigation of the interfacial fracture of barite rocks. The interfacial fracture toughness of barite (BaSO_4) rocks is studied using Brazil disk testing. This is used to determine the mode mixity dependence of the interfacial fracture toughness at mode mixities between mode I and mode II conditions. The crack/microstructure interactions associated with the measured resistance-curve behavior are also elucidated along with the underlying toughening mechanisms (crack bridging and crack kinking/deflection), which are revealed using optical microscopy. These are then incorporated into toughening models for estimating the fracture toughness/resistance-curve behavior at different mode mixities. The results show that the fracture toughness of barite rocks increases with increasing mode mixity. The implications of the results are then discussed for the applications of barite in oil and gas drilling, radiation shielding and aggregate materials.

1. Introduction

Barite (BaSO_4) is a heavy mineral that is widely utilized as a weighting agent in drilling fluids, in radiation shielding concretes, and as aggregate in specialized construction materials. Its mechanical reliability under loading is important in these applications, where crack/fracture initiation and propagation often occur at interfaces such as grain boundaries or between barite and secondary mineral phases. Fracture toughness is a key parameter for assessing a material's resistance to crack propagation [1]. While considerable attention has been devoted to fracture mechanics of ceramics and composites [2], research on the interfacial fracture toughness of geological minerals has been limited [3].

An examination of the published literature reveals a scarcity of information on mineral toughness, with the review by [4] providing a useful yet limited set of data [3]. Barite rocks are one of such

geomaterials. The weak interfaces between mineral grains can act as preferred crack paths, leading to premature material failure in structural and industrial applications. The initiation and propagation of failure in intact rock are a matter of fundamental importance in rock engineering. At low confining pressures, tensile fracture initiates in samples at 40–60% of the uniaxial compressive strength. Furthermore, at higher loads, the incidence of cracking increases, along with the occurrence of shear localization and macro-scale shear failure [5].

Fracture toughness (K_{IC}) is a key parameter used for measuring rock's resistance to crack propagation. This term is important in hydraulic fracturing operations for hydrocarbon reservoirs [6,7]. Luo et al. [8] conducted a sensitivity analysis of rock mechanical parameters and fracturing parameters at different scales using a dimensionless analysis method. This study reported that low-toughness samples can reproduce the initiation and propagation of field fractures in experiments. For barite rocks, the integrated laboratory experiments with reactive

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transport modelling of geochemical mechanisms governing barite (BaSO_4) scale formation in unconventional shale reservoirs by Esteves et al., [9] revealed that barite precipitation is strongly influenced by pH during hydraulic fracturing. Based on the findings, a predictive model that is capable of simulating scale formation under varying reservoir conditions was developed to enhance the understanding and mitigation of barite scaling in shale formations, thereby improving the effectiveness of scale-control strategies during oil and gas production, thereby improving the effectiveness of scale-control strategies during oil and gas production.

Wellbore strengthening and sealing technology has increasingly emerged as a key approach to enhancing the pressure-bearing capacity of formations. It is intended to increase the fracture pressure of formations prone to leakage and to address drilling fluid leakage under narrow safety density window conditions [10]. Rock fractures propagate through three principal modes, including Mode I (tensile opening), Mode II (in-plane shear), and Mode III or mixed-mode (out-of-plane shear) [11]. Although fractures encountered in engineering applications commonly propagate under mixed-mode loading [12], Mode I fracture toughness (K_{IC}) remains the most widely investigated parameter because rocks are more susceptible to tensile failure than shear failure [13]. The brittleness and lithological heterogeneity of interbedded sand-mud formations are inherent factors triggering wellbore instability [14,15].

Various experimental techniques have been developed to determine K_{IC} , which are broadly categorised into non-core and core-based methods. Non-core methods are the three-point and four-point bending tests, which require relatively large rectangular specimens that are often difficult to obtain from rock formations [16]. Core-based methods on the other hand, are more widely adopted because they utilise cylindrical core specimens that are easier to prepare and are particularly suitable for deep rock investigations. The core-based methods recommended by the ISRM include the Chevron Bend (CB) and Short Rod (SR) methods [17], the Cracked Chevron Notched Brazilian Disc (CCNBD) method [18], and the Semi-Circular Bend (SCB) method [19]. For the CB and SR methods, fracture toughness can be determined by recording either the peak load alone (Level I test) or both the load and displacement (Level II testing) to account for the nonlinear behaviour of rocks [17]. Nevertheless, direct determination of K_{IC} remains challenging due to limitations in specimen availability, demanding sample preparation procedures, and the cost and complexity of experimental testing.

Consequently, predictive models based on readily measurable rock properties have emerged as practical alternatives for estimating fracture toughness when laboratory testing is not feasible [20–22]. Ye et al. [23] reported that during the drilling process, examining changes in rock mechanics characteristics can effectively guide drilling and prevent well wall destabilisation accidents. Similarly, Haiyan Zhu [24] established a predictive model of rock mechanics parameters based on the results of uniaxial compressive strength (UCS), drillability, hardness, plasticity, and abrasiveness tests of cores from complex formations to improve the drilling rate in the western Nanhai oil field. Overall, dynamic unloading during drilling leads to the expansion of micro-fractures and the deterioration of mechanical parameters of rock masses [25–27]. Research on drilling fluid invasion further enriches the multi-factor coupling theory of wellbore instability [28–30].

In any case, the propagation of cracks can be studied in barite and other rocks, using fracture mechanics approaches [3]. Since this can occur over a wide range of mode mixities (between pure mode I and pure mode II conditions), the Brazil disc fracture mechanics specimen offers a simple approach for the study of interfacial and bulk fracture, using relatively small specimens with notches that can be oriented to change the mode mixities between pure mode I and pure mode II.

Brazil disc fracture testing has been successfully applied in brittle materials such as ceramics and perovskites, and its extension to barite rocks provides a pathway to quantify its interfacial reliability [31,32]. Interfacial fracture (due to crack initiation and propagation at grain

boundaries and mineral interfaces) has also been shown to occur in these regions due to cracks that can exist in polycrystalline and multiphase rocks. These can grow and coalesce during loading which can ultimately lead to catastrophic failure [33].

Furthermore, the ability of a material to resist cracking can be quantified by modeling the toughening mechanisms [3,34] ahead or behind the crack tip. These can result in resistance-curve (R-curve) behavior in rocks and their interfaces, as cracks extend along the bulk regions or along their interfaces with other materials. Hence, beyond the onset of crack initiation (which is often associated with limited underlying toughening), the growth of cracks is often resisted by toughening mechanisms that can occur either behind or ahead of the crack-tip [35, 36].

Furthermore, the interfaces between different mineral phases may exhibit mismatches in elastic modulus, thermal expansion, or chemical bonding, leading to stress localization under mechanical or thermal loading. Cracks, therefore, tend to nucleate at these interfaces, either from mechanical stress, thermal mismatch, or chemical weakening. They may, therefore, propagate along the interfaces or within adjacent bulk materials, depending on the relative values of their interfacial fracture toughness and their bulk fracture toughness levels, as well as their crack path selection criteria [35,36]. This leads to intergranular fracture, where cracks follow grain boundaries, or in some cases trans-granular fracture, where cracks cut through grains, when the interfaces are stronger.

In the case of barite, which is often injected into oil and gas wells during drilling operations, there is a need for fracture mechanics approaches to estimate critical conditions for modeling of interfacial and bulk fracture. Since this can occur over a range of mode mixities, there is a need for practical approaches for the measurement of interfacial fracture conditions over a range of mode mixities. Although this can be achieved using a range of interfacial fracture geometries [37], the Brazil disk specimen geometry offers the most convenient and compact geometry for the studying of interfacial fracture in rocks and a wide range of brittle materials and interfaces [32,38]. In-situ observations of the sides of Brazil disk specimen geometries have also been used to identify the crack/microstructure interactions that give rise to toughening and resistance-curve behavior in brittle materials and their interfaces.

Thus, in this study, Brazil disk specimens are used to study the effects of mode mixity on the interfacial fracture toughness of barite rocks. This is elucidated over a range of mode mixities between pure mode I and pure mode II. The underlying crack/microstructure interactions are also elucidated using an optical microscopy. Toughening mechanisms are then identified (ahead or behind the crack-tip). These are incorporated into fracture mechanics models for estimating of toughening and resistance-curve behavior. The implications of the results are then discussed for the design of robust barite interfaces that are associated with oil and gas drilling, and the design of barite aggregate structures.

2. Theory

2.1. Crack driving forces

A fracture mechanics approach was used in this study [35]. This uses the Energy Release Rate, G , or the stress intensity factor, K , to characterize the crack driving force. In the case of interfacial cracks, where the crack-tip fields are complex, the interfacial fracture toughness depends on the mode mixity (the ratio of mode II to mode I crack driving forces). In the case of the Brazil disc specimen geometries, the cracks are subjected to both opening (Mode I) and shearing (Mode II) forces, which influence the crack paths and crack growth rates [39].

In the case of the mixed-mode crack-tip field of the Brazil disc specimen geometry, the model I stress intensity factor is given by [31, 32]:

$$K_I = f_I \sigma(\pi l)^{-1/2} \quad (1)$$

where f_I is a function of the loading angle, θ , and relative crack length, (a/r) , that are dimensionless, l is the crack length, and σ is the remote stress applied to the specimen (Fig. 3a). The mode I energy release rate (for the Brazil disk specimen) is given by:

$$G = G_I = \frac{1}{E^*}(K_I^2) \quad (2)$$

where E^* is the plane strain Young's modulus of the sample. Also, the stress intensity factor for mode II is given by [31,32]:

$$K_{II} = f_{II}\sigma(\pi l)^{-1/2} \quad (3)$$

where f_{II} is a function of the loading angle, θ , and the mode II energy release rate is given by:

$$G = G_{II} = \frac{1}{E^*}(K_{II}^2) \quad (4)$$

The total energy release rate, G_{Total} , for mode I and mode II is given by:

$$G_{Total} = G_I + G_{II} = \frac{1}{E^*}(K_I^2 + K_{II}^2) \quad (5)$$

where G_I and G_{II} are the total strain energy release rates for mode I and mode II. Also, the stress intensity factors for mode I and mode II can be calculated using 6 and 7. These give:

$$K_I = \sigma\sqrt{\pi a}\cos^2\left(\frac{\theta}{2}\right) \quad (6)$$

$$K_{II} = \sigma\sqrt{\pi a}\sin\theta\cos\left(\frac{\theta}{2}\right) \quad (7)$$

where σ is the remote stress applied to the Brazil disk sample, a is the crack length and θ is the loading angle. The remote stress applied to the Brazil disk sample (σ) is given by:

$$\sigma = \frac{P_{max}}{\pi r t} \quad (8)$$

where P_{max} is the maximum applied load (that causes fracture during the testing), r is the radius of the disk and t is the thickness of the disk. The mode mixity of the Brazil disk specimen (for biomaterials) is given by:

$$\Psi = \tan^{-1}\left(\frac{K_I}{K_{II}}\right) + \omega + \varepsilon \ln\left(\frac{\hat{L}}{h}\right) \quad (9)$$

where $\hat{L}$ is a length to define the loading phase and h is the thickness of the layer, ω is the shift due to elastic modulus mismatch in bi-material pairs. It is a function of Dundurs parameters, α , β and ε , which are given by:

$$\alpha = \frac{\frac{1-\nu_1}{\mu_1}}{\frac{1-\nu_1}{\mu_1}} \quad (10)$$

$$\beta = \frac{1}{2} \frac{\frac{1-2\nu_1}{\mu_1}}{\frac{1-\nu_1}{\mu_1}} \quad (11)$$

$$\varepsilon = \frac{1}{2} \ln\left(\frac{1-\beta}{1+\beta}\right) \quad (12)$$

where μ is the shear modulus and ν is the Poisson's ratio for each layer. Also, the shear stress and Poisson's ratio values for barite rocks (that were used in this work) were 21.01 GPa and 0.33, respectively.

In the case of the barite rock material, ω is the elastic modulus mismatch of the bi-material pairs (in Eq. 9) is not applicable. Thus, the mode mixity for barite rock is given by:

$$\Psi = \tan^{-1}\left(\frac{K_{II}}{K_I}\right) + \varepsilon \ln\left(\frac{\hat{L}}{h}\right) \quad (13)$$

2.2. Toughening mechanisms

Interfacial fracture toughening models incorporate mechanisms such as plastic deformation, crack bridging, microcracking, and interface roughening to reduce the crack driving forces. Row and zone models, originally introduced by [40], have been used to evaluate the levels of crack-tip shielding corresponding to the observed crack profiles.

In this work, we use mixed-mode fracture models, to account for the combined effects of opening (Mode I) and shearing (Mode II) forces, and the zone model does not incorporate frictional effects, with both the deflection angle (β) and friction angle (θ) set to zero, as noted by [31,38,41]. In this model, shear stresses and displacements are treated as elastic and are assumed to take after those found in a linear arrangement of micro-cracks. In the zone model, forces under Mode I and Mode II loading conditions are analysed to estimate crack-tip shielding levels, as shown in Fig. 1.

The zone model, introduced as a secondary approach by Evans [40], uses a simplified representation of the crack profile, as illustrated in Fig. 1. According to Eqs. 15 and 16, several parameters define the behavior of the zone model: E represents Young's modulus, L is the length of the zone, H denotes the height of the interface step, D is the length of each facet, and l refers to the spacing between the centers of these micro-crack facets. These variables collectively influence the mechanical response of the interface under stress.

The zone model provides an easy way of estimating the toughening increment, which is the increase in fracture resistance due to crack-tip shielding. This ratio helps in understanding elastic interactions within the crack zone and the contribution of such interactions to the material's toughness. The change in toughening estimated by the zone model [42] is given by:

$$\frac{\Delta G}{G} = \frac{\tan^2\Psi\{1 - k[\alpha_0(1 + \tan^2\Psi)(\frac{\Delta G}{G} + 1)]\}}{1 + \tan^2\Psi} \quad (14)$$

where k is a function of α_0 that can be obtained from $1/\lambda$ Table 1 in [31,32,42] and α_0 is a material parameter given by:

$$\alpha_0 = \frac{\pi E H^2 / l G_0}{32(1 - \nu^2) \ln(1 / \sin(\frac{\pi D}{2l}))} \quad (15a)$$

Also, α_0 can be calculated using Eq. 15b, which gives: [31]

$$\alpha_0 = \frac{E H / G_0}{(1 - \nu^2)} \quad (15b)$$

$$\frac{D}{l} = \frac{1}{2}, \quad \frac{H}{l} = \frac{1}{2} \quad (16)$$

where H denotes the height of the interface step, D is the length of each facet, and l refers to the spacing between the centers of these micro-crack facets.

Also, the contact zone dimension is given by:

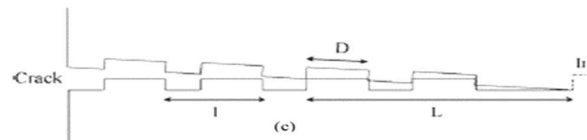


Fig. 1. Zone model diagram (Adapted from [6]).

Table 1

Geometry and Dundur's parameters for barite rock. These are values calculated using relevant equations and theory, as discussed.

Geometry of Sample	Thickness of disk (mm)	Diameter of disk (m)	Radius of disk (mm)
Dundurs Parameters of Barite Rock	12	36	18
α	1	2	3
ϵ	1.0	0.51	-0.179

$$\chi = \frac{EH}{G_0} \quad (17)$$

The geometry of the Brazil disc sample used and the Dundur's parameters of the barite rock (calculated using 10, 11, 12, and 13) are presented in Table 1

For large values of χ , the toughening can be obtained from Eq. 18 to be given by:

$$G = G_0(1 + \tan^2 \Psi) \quad (18)$$

2.3. Crack path selection

Crack path selection is a critical aspect of fracture mechanics. It is influenced by the relative ratio of the interfacial fracture toughness to the matrix fracture toughness and the mode mixity [35,40]. Hence, if the interfacial fracture toughness is low, interfacial cracking is more likely to occur initially. However, changes in the mode mixity may result in transitions from interfacial cracking to cracking in the bulk/substrate layers. Mixed-mode loading often leads to complex crack paths, including crack kinking or branching. Surface treatments, such as chemical adhesion promoters, and geometric factors, such as layer thickness and curvature, also affect crack trajectory. However, computational tools, such as Finite Element Analysis (FEA), and Extended Finite Element Method (XFEM) are needed to simulate the crack growth under such conditions.

3. Materials and experimental procedures

The barite materials used in this work were obtained from the Ibi Local Government area of Taraba State, Nigeria, West Africa. Brazil disc specimens with diameters of 36 mm were machined at the African University of Science and Technology (AUST) in Abuja, Federal Capital Territory (FCT), Nigeria. These were then tested in an electromechanical testing machine (Instron Model 3345, Instron, Norwood, Canton, MA) at AUST. Fig. 2 shows the experimental setup of the Sony high-definition camera (Sony 4k Model, PXW-Z150 XAVC/XDCAM, Minato, Tokyo, Japan) during the experimentation.



Fig. 2. The laboratory experimental setup for Brazil disc experiment for real-time imaging of the barite rock disc specimens.

The barite rocks were cut and polished using a Buehler Eco-met 300 Pro system with different-sized grit paper that were mounted on an adjustable rotating disc at AUST. The notches were then fabricated with a handheld saw blade with a thickness of 1 mm and a height of 5 mm. In this way, notches with a length, L , of 4 mm and a width, W , of 2 mm, were introduced into the samples. Further details of the sample geometry are summarised in Table 1.

Similarly, the number of specimens tested for each loading angle is 2. Similarly, each of the IB2 and IB3 barite rock samples were tested or loaded, which is ten (10) IB2 barite rock samples and ten (10) IB3 barite rock samples tested in all for loading angles 0° , 3° , 7° , 13° and 23° . Hence, a total of twenty (20) specimens or barite rock samples were tested and examined during the Brazilian experiments.

The fracture toughness tests of IB2 barite rock samples by Brazil disc specimens (Fig. 3a) at the rate of 0.09 mm/min ensures the compressive loading of rock samples at different angles, using an Instron 3345 electromechanical testing machine (Instron, Norwood, MA, USA), which is equipped with a 5 kN load cell. The mode mixities were controlled by turning specimen notches orientations relative to the vertical axis. Hence, from Eq. 13, these correspond to average mode mixity angles (Ψ) of 0.73° , 3.74° , 7.69° , 13.49° and 22.59° were obtained and a standard deviation of 0.005, 0.01, 0.01, 0.005 and 0.105 were observed at each loading angle for the set A and Set B of the IB2 samples respectively. In the case of the IB3 rock sample, the mode mixity angles (Ψ) of 0.72° , 3.75° , 7.70° , 13.50° and 22.43° and a standard deviation of 0.005, 0.005, 0.025, 0.015 and 0.000 were obtained at each loading angle for the set IB3 A & IB3 B respectively.

The growth of cracks (from each of the notch tips) were monitored continuously (using a digital camera) during incremental stages of crack growth at different loads. The length of the cracks obtained from the images were measured using Gwydion software package. Furthermore, resistance-curves were obtained from plots of energy release rates versus crack extension. Data was extracted during the testing from the data interface of the electromechanical testing machine and computed to characterise the variabilities in the measured initiation toughness values and the resistance-curves. A combination of digital cameras (Sony 4k Model, PXW-Z150 XAVC/XDCAM, Minato, Tokyo, Japan) was used to study the material and the crack/microstructure interactions at different stages of crack growth respectively. Hence, the statistical analysis of the data ensures the evaluation of variability and the standard error calculated from the standard deviation computed using data from repeated measurements).

While the SEM-EDS were used to study the microstructural composition of the rock samples. The Images of Brazil disk samples are presented in Fig. 3a & b.

4. Results and discussion

4.1. Mode mixity dependence of fracture toughness

The estimated toughening levels (for the zone model), $G = \Delta G + G_0$

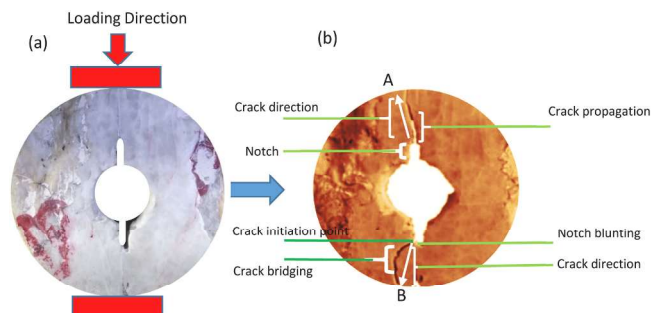


Fig. 3. Images of Brazil disk sample, (a) before testing and (b) after testing during the Brazilian disc experiments.

(J/m²), are presented in Table 2 along with the corresponding initiation toughness values and mode mixity levels (calculated using Eq. 13) for the tested loading angles of 0°, 3°, 7°, 13° and 23° applied to samples IB2 and IB3”.

4.2. SEM-EDS analysis of samples IB2 and IB3 mineral interfaces

For the geological and structural distinction of the two samples, the SEM-EDS analysis of the samples are presented in Fig. 4.1. In addition, a brief explanation has been included describing the geological and structural distinction between the IB2 and IB3 interfaces to improve the clarity of the experimental methodology.

The SEM-EDS images of the barite rocks samples studied are presented in Fig. 4.1

The SEM micrograph of IB3 reveals a relatively dense and homogeneous microstructure with uniformly distributed fine particles and minimal visible porosity. This compact morphology indicates effective particle packing and strong inter-particle bonding, suggesting the formation of a well-consolidated matrix. The absence of pronounced microcracks and large voids further reflects good structural integrity, which can contribute positively to the mechanical performance of the material and subsequent resistance to failure under loading.

The corresponding EDS analysis shows that IB3 is primarily composed of oxygen (42.82 wt%), carbon (24.82 wt%), and silicon (20.78 wt%), indicating that silicate phases dominate the matrix. Minor amounts of aluminium (5.73 wt%), barium (5.01 wt%), iron (1.61 wt %), calcium (0.45 wt%), potassium (0.35 wt%), sodium (0.34 wt%), and magnesium (0.04 wt%) were also detected. The presence of silicon and aluminium confirms the occurrence of aluminosilicate minerals, while barium indicates the incorporation of barite within the matrix. The relatively high carbon content is likely associated with residual carbonaceous material, or the conductive carbon coating applied during SEM sample preparation.

In contrast, the SEM image of IB2 displays a rougher and more heterogeneous surface characterised by particle agglomeration and interconnected clusters. As compared to IB3, the microstructure contains more irregular features and intergranular voids, indicating a less compact matrix. Such structural characteristics may facilitate crack initiation and propagation under mechanical loading. However, the continuous network of particles suggests that adequate bonding still exists among the constituent phases. The EDS spectrum of IB2 also supports the structural characteristics, as mentioned. Several non-barium elements such as oxygen, carbon, silicon, and traces of

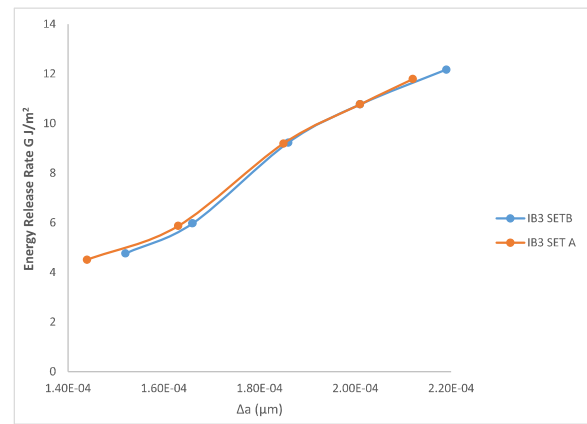


Fig. 4.1. SEM-EDS (combined scanning electron microscopy-energy dispersion spectroscopy) of Samples IB2 and IB3. The images indicate the elemental mapping, morphology, and electron-energy eds peak match for each element identified.

aluminium, iron, calcium, potassium, sodium, and magnesium are indicated in addition to higher barium, which is the dominant element. The comparatively higher barium content indicates a greater concentration of the barite phase in IB2, whereas the lower silicon content suggests a corresponding reduction in the aluminosilicate matrix. This compositional variation confirms that the distribution of mineral phases differs between the two samples.

Overall, the SEM-EDS results demonstrate that both IB2 and IB3 consist of an aluminosilicate matrix containing dispersed barite particles. However, IB3 exhibits a denser and more uniform microstructure, while IB2 is characterised by a rougher and more agglomerated morphology caused by a higher concentration of barium-rich particles. Similarly, the XRD peaks and the quantitative XRD data (published as part of the preliminary results), indicate that the minerals grain size is between 0.058 μm and 0.075 μm for IB2. The values are between 0.0146 μm and 0.038 μm for IB3. Using the Scherrer Equation, the average grain size was calculated as 57.01 nm (0.057 μm). These microstructural and compositional differences are expected to influence the engineering behaviour at different loading conditions, and the beneficiation potential of the materials. The compact structure of IB3 can enhance mechanical stability, and the elemental mapping provides an estimate of the economic worth and industrial application.

Table 2

Experimental and statistical data for barite rock samples IB2 and IB3 for Set A & B samples used in zone model computation at different mode mixity angles.

Average Mode Mixity Angles (Ψ°)			G = ΔG + G ₀ (J/m ²)		
IB2 SET A G ₀ = 3.0 J/m ²	IB2 SET B G ₀ = 2.5 J/m ²	Standard Deviation	IB2 SET A	IB2 SET B	Standard Deviation
1	0.73	0.005	3.000	2.500	0.250
2	3.75	0.010	3.013	2.510	0.252
3	7.70	0.010	3.060	2.560	0.252
4	13.49	0.005	3.170	2.640	0.265
5	22.48	0.105	3.510	3.550	0.200
Average Mode Mixity Angles (Ψ°)			G = ΔG + G ₀ (J/m ²)		
IB3 SET A G ₀ = 4.76 J/m ²	IB3 SET B G ₀ = 4.20 J/m ²	Standard Deviation	IB3 SET A	IB3 SET B	Standard Deviation
1	0.72	0.005	4.760	4.200	0.280
2	3.75	0.005	4.780	4.220	0.280
3	7.67	0.025	4.850	4.280	0.285
4	13.48	0.015	5.030	4.440	0.295
5	22.48	0.000	5.570	4.920	0.325

4.3. Resistance-curve behavior

The resistance-curve behavior obtained for the different conditions is summarized in Figs. 4a and 4b. These show that the set A samples generally exhibit stronger resistance curve-behavior than the set B samples.

The resistance curves for SET A and SET B for IB2 show how the materials respond to crack growth in terms of energy release rate. Both sets exhibit R-curve behavior, where fracture resistance initially rises with crack extension before reaching a peak. This trend reflects the balance between energy driving crack propagation and the material’s ability to resist fracture through mechanisms like grain bridging or crack deflection. For SET A, fracture resistance increases rapidly from a low initial value of about 2.4 J/m², reaching a maximum of approximately 21.1 J/m² at a crack extension of about 2.4 × 10⁻⁴ μm. However, after this peak, resistance falls showing that once the material reaches its maximum toughness, it quickly loses stability and becomes more prone to brittle fracture. This behavior suggests that SET A provides good resistance during early crack growth but is less effective at sustaining toughness over longer crack propagation. SET B, by contrast, exhibits a steadier rise in resistance, starting at about 5.2 J/m² and gradually increasing to a higher maximum of 23.6 J/m² at 2.6 × 10⁻⁴ μm.

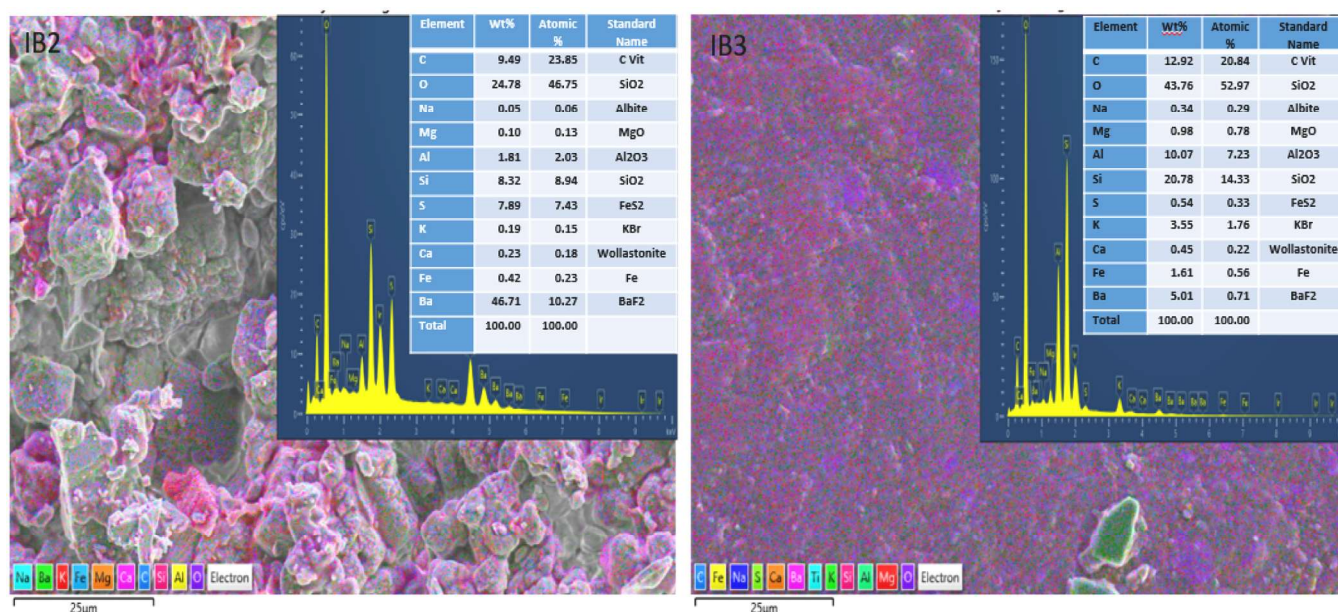


Fig. 4a. IB2 Resistance Curve Set A and Set B.

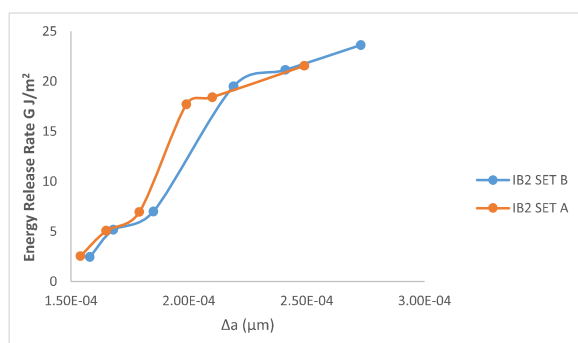


Fig. 4b. IB3 resistance curve Set A and set B.

Importantly, it maintains its resistance over a broader crack extension range. This indicates that SET B has better overall fracture resistance, resisting crack propagation for longer than SET A. Fig. 4b shows the resistance curve for IB3 set and SET B.

Fig. 4b shows IB3 Resistance Curves for SET A and SET B, comparing how each side of the material resists crack growth as the crack extension (Δa) increases. Both curves exhibit a typical R-curve pattern where fracture resistance, measured as energy release rate (G), rises with crack extension. Initially, both sets have similar resistance values ranging from 4.0 to 5.0 J/m², with SET B slightly higher. As the crack grows, resistance increases steadily in both cases, reaching nearly the same maximum of about 12 J/m² near $\Delta a \approx 2.1 \times 10^{-4}$ μm. Beyond this point, however, the behaviors diverge indicating a more brittle response once maximum toughness is reached. In contrast, SET B maintains resistance for a longer period, suggesting greater stability during extended crack growth.

The resistance curves from sample IB2 and IB3 both show how fracture resistance evolves with crack growth, but they differ in overall toughness levels. In IB2, both SET A and SET B achieved much higher peak resistance values, with SET A reaching around 21.0 J/m² and SET B peaking even higher at about 23.6 J/m². SET A showed a rapid increase in resistance but lost toughness abruptly after the peak, indicating brittle behavior.

Also, SET B increased more steadily and maintained higher resistance over a longer crack extension showing greater stability against

crack propagation. In IB3, the overall resistance levels were noticeably lower, with both SET A and SET B peaking at around 12 J/m², roughly half the maximum values observed in IB2. Although SET A again displayed a lower resistance compared to SET B which maintained fracture resistance for a longer duration suggesting that it remains the less tough material under extended crack growth.

The patterns of behavior in IB3 mirrored those in IB2, but with reduced peak toughness values and more bridging in IB2 samples. These results show that for barite rock, the R-curve shapes particularly the SET A vs. SET B differences are consistent with fracture governed by grain boundary and mineral to mineral interfaces in a brittle, cleavage-prone sulfate. The higher peak toughness in IB2 (20 J/m² to 24 J/m²) suggests stronger toughening mechanisms e.g., more effective grain bridging, crack deflection at barite-to-barite boundaries, or tougher barite to other associate mineral contacts (e.g., quartz/calcite/albite).

In contrast, the lower peaks in IB3 (10 J/m² to 12 J/m²) indicate fewer or weaker barriers to crack advance, implying impurity less interfaces, smaller or less interlocked grains, or prior micro-damage that reduces bridging. The repeated pattern of SET A rising fast, SET B rising steadily maps onto microstructural contrasts: SET A likely has weaker or more planar boundaries that promote intergranular runaway once peak is reached (brittle post-peak), whereas SET B likely contains rougher, more tortuous interfaces, second-phase pinning, or residual compressive stresses that keep the crack path deflected and bridged over a longer crack extension (Δa). Furthermore, Figs. 5a–5d present the plot of energy released rate vs mode mixities of the analyzed samples.

Fig. 5a shows the plot of IB2 energy release rate versus mode mixity for SET A with a clear dependence of fracture resistance on the type of loading. At low mode mixity angles (around 3.6°), which correspond closely to pure opening mode (Mode I), the energy release rate is very low at about 2.3 J/m². This indicates that the material is most vulnerable to cracking when pulled apart directly, suggesting that grain boundaries or mineral interfaces in the barite structure are the weakest against tensile stresses that open cracks. As the mode mixity angle increases into the mid-range (8.6°–13.6°), fracture resistance rises gradually, reaching about 6.0 J/m². This transition region shows that the addition of shear stresses near (Mode II) to the loading increases the difficulty of crack propagation. The crack path is no longer opening easily, instead it involves some level of sliding or deflection along grain boundaries, requiring more energy to sustain growth. This is evident in Fig. 3b from the crack bridging. This behavior highlights how

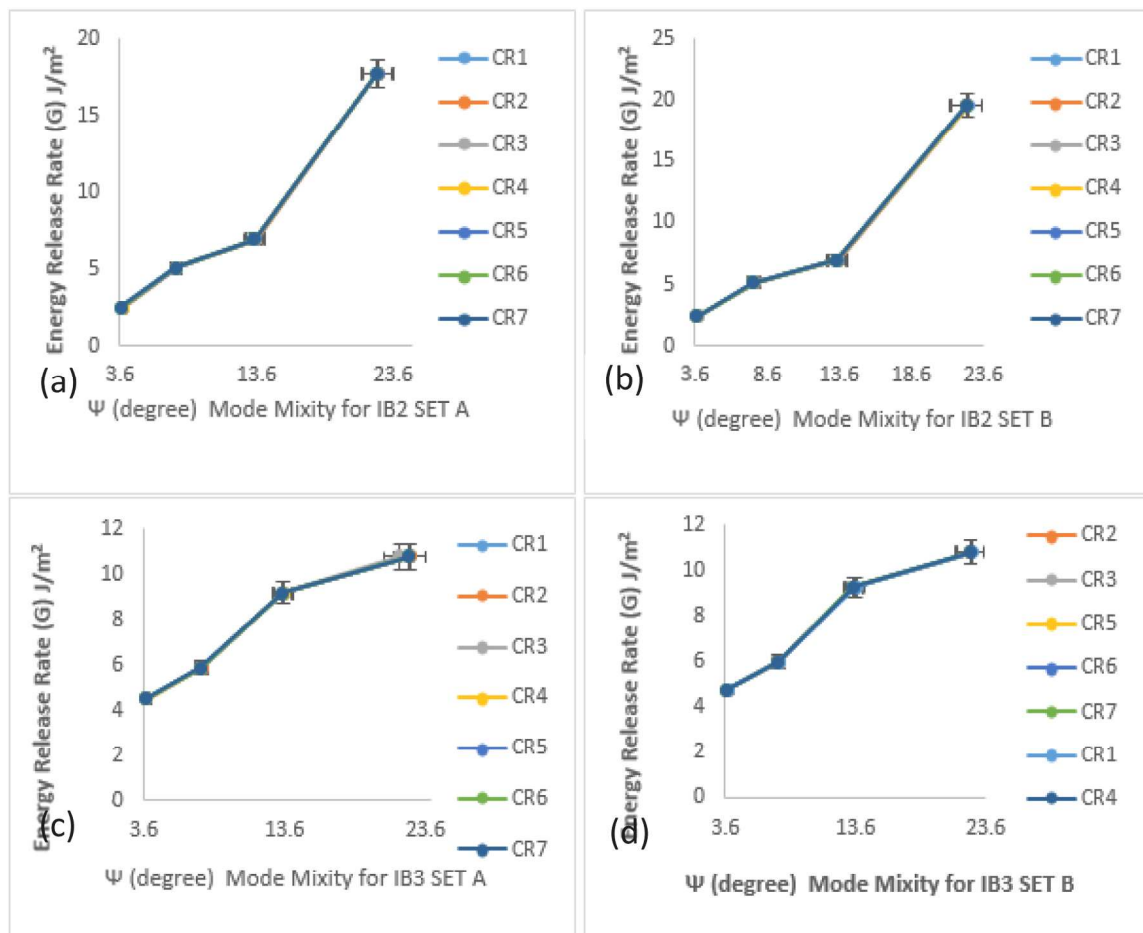


Fig. 5. a-5d: Energy release rate vs mode mixities. (Fig. 5a) IB2 energy release rate vs mode mixities set A and (Fig. 5b). IB2 energy release rate vs mode mixities set B. Fig. 5c. IB3 energy release rate vs mode mixities set A. (Fig. 5d) IB3 energy release rate vs mode mixities set B.

microstructural features, such as boundary roughness or interlocking grains, contribute to resisting crack advance under mixed loading.

At higher mode mixities (18.6°–23.6°), fracture resistance increases sharply, peaking near 17.0 J/m². This shows that, when shear dominates, the barite rock interfaces and boundaries dissipate much more energy during fracture, likely through mechanisms such as crack deflection, intergranular sliding, and frictional resistance. Overall, the results suggest that barite rocks are significantly tougher under combined tensile and shear stress than under pure opening, which has important implications for their behavior in natural fracturing, drilling environments, and industrial grinding. This indicates that the material's interfaces or grain boundaries are more resistant to crack growth when shear stresses contribute, likely due to increased energy dissipation mechanisms (e.g., sliding, rough boundary interactions). Thus, the fracture behavior of this barite system is highly sensitive to mode mixity, becoming tougher under combined opening and shear loading.

Fig. 5b presents plots of energy released rate vs mode mixity for set of the IB2 specimens. It illustrates how the energy release rate (G) varies with mode mixity angle (Ψ°) across different test conditions labeled. Each curve shows, a consistent upward trend, indicating that as the mode mixity increases from 3.6° to 23.6° and the energy available for crack propagation also rises.

Fig. 5c shows IB3 resistance curves which indicate that fracture resistance in Set A depends strongly on mode mixity. At low mode mixity angles (3.6°), where the loading is dominated by opening stresses (Mode I), the energy release rate is relatively low at about 4.0–5.0 J/m². This indicates that the material is most vulnerable when cracks are driven mainly by tensile opening, as the interfaces and grain boundaries

provide less resistance under this loading condition. As the mode mixity increases to around 8.6° and 13.6°, fracture resistance rises steadily, reaching values close to 9.0 J/m². This suggests that the addition of shear stress (Mode II) makes crack propagation more difficult, likely because shear promotes mechanisms such as crack deflection, grain boundary sliding, or increased frictional resistance at the interfaces. These mechanisms require more energy to continue crack growth, thereby increasing the material's toughness.

At higher mode mixities, particularly between 18.6° and 23.6°, the fracture resistance continues to increase but begins to plateau around 10 J/m² to 11 J/m². This indicates that the material reaches a saturation point where additional shear contribution no longer significantly raises resistance. Overall, IB3 set A demonstrates that fracture resistance improves with mixed-mode loading, but levels off under shear-dominated conditions. Also, Fig. 5d shows the IB3 resistance curve for set B highlighting the clear relationship between fracture resistance and mode mixity.

From Fig. 5d, at low mixity angles (3.6°), where the loading is dominated by opening stresses (Mode I), the energy release rate ranges from 4.0 J/m² to 5.2 J/m². This low resistance highlights that the material is weakest under tensile opening, with cracks propagating more easily when shear contribution is minimal. As the mode mixity increases to about 7.6°–13.6°, the fracture resistance rises steadily, reaching values close to 9.0 J/m². This reflects the role of shear stress in hindering straightforward crack opening, as mixed loading promotes mechanisms such as crack deflection, interactions along grain interfaces. These processes demand more energy for the crack to grow, thereby enhancing the material's toughness.

At higher mixity levels, around 21.6° , the resistance continues to increase gradually and stabilizes between 10 J/m^2 to 11 J/m^2 . Overall, IB3 set B demonstrates that fracture resistance improves progressively with shear contribution, but like set A, it eventually plateaus under high shear-dominated conditions, showing a saturation in toughening mechanism. The IB3 resistance curves for both set A and set B demonstrate a strong dependence of fracture resistance on mode mixity. At low mode mixity angles (around 3.6° to 5.6°), where loading is dominated by opening stresses (Mode I), the energy release rate is relatively low at about $4\text{--}5 \text{ J/m}^2$. As mode mixity increases into the intermediate range (8.6° to 13.6°), the resistance rises steadily, with values approaching about 9.0 J/m^2 for both sets. At higher mode mixities (18.6° to 23.6°), both set A and set B reach their peak resistance values in the range of 10 J/m^2 to 11 J/m^2 .

4.4. Comparison of measured and predicted resistance-curves

A comparison of measured and predicted resistance-curves is presented in Figs. 6a–d. These show that the model reproduces the general trends observed in the experimental data. However, it fails to adequately capture the behavior under conditions of high mode mixity and elevated energy release rates in sample IB2 set A and set IB3 set A, as presented below.

The results presented in Fig. 6a compare the experimental results and the simplified zone model predictions for the total energy release rate G_{Total} in IB2 Set A as a function of mode mixity angle (Ψ). From the plot, it is evident that the experimental energy release rate starts at around 3.0 J/m^2 for low values of (Ψ) and gradually increases, reaching approximately 3.5 J/m^2 at higher mode mixity angles. In contrast, the simplified zone model predicts a constant energy release rate of about

3.0 J/m^2 , independent of Ψ . At lower mixity angles ($0^\circ\text{--}10^\circ$), the experimental values are close to the zone model predictions, and their error bars suggest the differences are within experimental uncertainty. However, as the mode mixity increases beyond 15° , the experimental data consistently trend upward, showing a clear dependence of G_{Total} on (Ψ), while the zone model predictions fail to capture this behavior. This divergence highlights the limitation of the simplified zone model, which neglects the effect of increasing shear contribution on the energy release rate. Overall, the results indicate that while the simplified zone model can provide a reasonable approximation under near-mode I conditions, it becomes less accurate at higher mode mixities. A more sophisticated fracture model would therefore be necessary to capture the increasing energy release rate observed experimentally.

Fig. 6b presents a comparison between experimental results obtained for IB2 Set B and the simplified zone model predictions of the energy release rate G_{Total} as a function of mode mixity angle (Ψ). Both datasets exhibit a similar upward trend, with increasing (Ψ) at around 2.4 J/m^2 for low mixity angles and reaching approximately 2.9 J/m^2 at higher values. Unlike Set A, the simplified zone model in Set B closely follows the experimental results across the entire range of (Ψ), with only minor deviations that remain within the experimental error margins. This indicates a good level of agreement between the model and the experimental data, suggesting that the simplified zone model is more effective in capturing the variation of fracture energy with mode mixity in this case. The consistency between the two curves implies that, for IB2 Set B, the simplified zone model provides a reliable representation of the experimental behavior, making it a more suitable predictive tool compared to its performance in Set A.

The IB3 comparison between experimental data and the simplified zone model for set A in Fig. 6c shows differences in how fracture

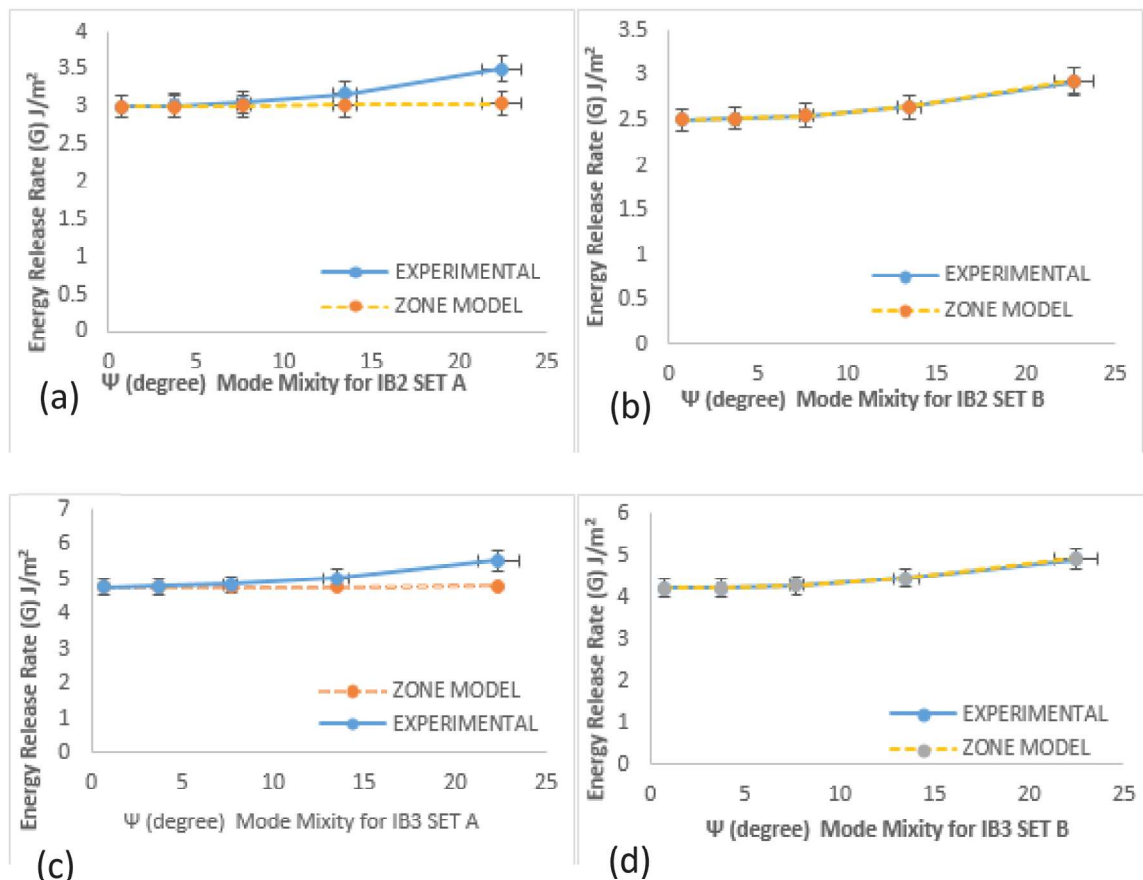


Fig. 6. A comparison of plots of experimental and predicted energy release rates obtained from the simplified zone model: (a) IB2 set A, (b) IB2 Set B, (c) IB3 Set A, (d) IB3 set B.

resistance varies with mode mixity. The experimental curve begins at about 4.8 J/m^2 under low mode mixity and shows a gradual increase as the mode mixity angle (Ψ) reaches nearly 6 J/m^2 at higher values around 23° . This upward trend suggests that, in practice, the material exhibits some degree of shear-driven toughening, where fracture resistance increases modestly as shear components contribute more significantly to the loading condition.

In contrast, the simplified zone model predicts a nearly constant energy release rate of about 4.8 J/m^2 to 4.9 J/m^2 across all mode mixity values. The flat line suggests that, within the model's assumptions, fracture resistance is not sensitive to the contribution of shear stresses and remains essentially fixed regardless of the loading condition. The discrepancies between the predicted and the experimental results in the toughening levels were also observed in the work of [31], ceramic/epoxy coated materials. The experimental findings demonstrate that IB3 set A exhibits measurable toughening under mixed-mode loading, even if the increase is moderate. Therefore, the simplified model may serve as a baseline estimate but does not fully capture the material's real fracture behavior, particularly in conditions involving significant shear.

From the results, the experimental approach rises with Ψ while the simplified zone model stays flat, or insignificantly rises in terms of barite's microstructure. Barite is brittle with pronounced cleavage and anisotropy; cracks under Near-Mode-I loading can open along relatively planar grain or cleavage interfaces with low resistance. As shear is added (higher Ψ), real interfaces are rough and mismatched; contact asperities engage [31], microcracks deflect, and grains slide or bridge. These mechanisms dissipate extra energy through frictional sliding, crack-tip shielding, and so the measured G_{Total} increases with Ψ . Also, the simplified zone model, by contrast, effectively assumes a constant interfacial toughness (mode-independent) and linear-elastic behavior with no friction, roughness, or bridging. It treats the interface as a flat, uniform plane with fixed normal/shear cohesion and therefore cannot capture anisotropy, grain-boundary morphology, or the activation of shear-dependent dissipation.

The plot for IB3 Set B in Fig. 6d illustrates the comparison between experimental measurements and the simplified zone model predictions of the energy release rate G_{Total} as a function of mode mixity angle Ψ . Both the experimental data and the model exhibit a consistent upward trend, initiating around 4.0 J/m^2 at low values of (Ψ) and increasing steadily to approximately 4.6 J/m^2 at higher mixity angles. The agreement between the experimental results and the model is very strong, with the two curves nearly overlapping across the entire range. This indicates that, for IB3 Set B, the simplified zone model accurately captures the dependence of fracture energy on mode mixity and providing a highly reliable representation of the experimental behavior.

Unlike in Set A, where the model underestimated the increase in G_{Total} , the results for Set B demonstrate the robustness of the model in replicating experimental trends under these conditions. The comparison highlights that the simplified zone model performs well and captures the trends across different material sets. However, it under predicts the fracture energy in IB2 Set A, especially at higher mode mixities, but shows excellent agreement with experimental data in both IB2 Set B and IB3 Set B. This suggests that the reliability of the simplified zone model is strongly dependent on the specific material system and loading conditions, being well-suited for some cases but inadequate for others.

Therefore, the simplified zone model is used in this study as a baseline approximation of interfacial fracture behaviour, assuming linear-elastic shear and displacement responses and neglecting frictional effects, crack deflection, and friction angles. Under these assumptions, the model provides a simple estimate of crack-tip shielding and the associated toughening increment. The model performs reasonably well where the interface response is dominated by mode-I loading or where the material response follows the assumed simplified elastic behaviour. This is demonstrated by the close agreement between predicted and experimental fracture resistance for IB2 Set B and IB3 Set B. However, its predictive capability becomes limited under higher mode mixity,

particularly for IB2 Set A and IB3 Set A, where the experimental fracture resistance increases with increasing shear contribution while the model remains nearly constant.

These discrepancies arise because the simplified formulation does not explicitly account for interface roughness, frictional sliding, crack bridging, anisotropy, grain-boundary morphology, or other shear-dependent dissipation mechanisms. Therefore, the model should be regarded primarily as a baseline approximation rather than a comprehensive representation of the actual mixed-mode fracture behavior.

5. Implications

5.1. Implications for drilling of barite rock-containing wells for oil and gas

The results of the fracture resistance tests have implications for drilling barite-containing wells in oil and gas operations. The findings show that barite rock is weakest under opening-dominated stresses (Mode I), where fracture resistance is very low. This means that when the near-wellbore environment is subjected to tensile stresses, such as those caused by excessive mud pressure or unfavorable stress concentrations, cracks can easily initiate along grain boundaries or interfaces. This vulnerability increases the likelihood of wellbore instability problems such as lost circulation, induced fractures, and breakouts if mud weight is not properly controlled.

In contrast, under mixed-mode or shear-dominated loading, barite exhibits much higher resistance, with fracture toughness rising sharply. This suggests that shear-related stresses, such as those generated during drilling or from natural in-situ stress conditions, make crack growth more difficult. While this improves wellbore stability under shear-dominated regimes, it also means that drilling tools will encounter higher resistance, resulting in slower rates of penetration and greater drill bit wear due to energy dissipation through mechanisms such as crack deflection, grain boundary sliding, and frictional resistance.

For hydraulic fracturing operations, the results suggest that fractures in barite zones will initiate readily under tensile stresses but will encounter increased resistance to propagation once the shear components dominate, potentially limiting fracture growth and altering fracture geometry. This makes barite-rich layers act as fracture initiation points but poor pathways for sustained fracture extension. Overall, these results highlight the mechanical sensitivity of barite-bearing formations: they are highly vulnerable to opening-mode cracking but significantly tougher under shear-dominated conditions. Effective drilling in such formations therefore requires careful mud weight management to prevent Mode I fracture initiation, the use of wear-resistant drill bits to withstand high shear resistance and tailored hydraulic fracturing strategies to account for the limited fracture propagation capacity of barite-rich intervals.

Also, the comparison between the experimental data and the simplified zone model has direct implications for drilling barite rock-containing wells in oil and gas operations. The key observation is that the simplified zone model works reasonably well in some cases (IB2 Set B, IB3 Set B), where its predictions closely follow the experimental results, but it fails in others (IB2 set A, IB3 set A), especially at higher mode mixities where shear-driven mechanisms significantly increase fracture resistance. This inconsistency highlights that the fracture response of barite rocks is highly dependent on its microstructural characteristics, including cleavage planes, grain boundary morphology, and anisotropy, which are not accounted for in the simplified zone model.

The implications of the inconsistent prediction at higher mode mixity can significantly affect drilling operations owing to the wide range of assumptions. For instance, predictive models used to estimate wellbore stability, fracture initiation, or lost circulation risks may not always be reliable if they assume constant fracture resistance independent of mode mixity. In real barite rock-bearing formations, fractures under near-mode-I loading will initiate more easily along weak planes, making the rock prone to crack opening and potential instability when mud

pressures induce tensile stresses.

However, as drilling introduces shear components, the actual resistance of the formation can increase due to mechanisms such as grain sliding, frictional resistance, and crack deflection, which the simplified model was unable to completely capture. This could lead to underestimation of the toughness of barite zones under mixed-mode stresses, resulting in misprediction of drilling behavior.

Fig. 7 shows the failure surface features of the materials resulting from the mechanical deformation of the barite rocks. The results identify the crack directions, bridging and other mode mixity fracture characteristics. Also, IB2 samples display sharp, straight cracks and smoother surfaces, indicating brittle fracture with limited energy absorption. In contrast, the IB3 samples exhibit rougher, more irregular cracks with signs of blunting and deflection, suggesting higher fracture resistance and tougher behaviour. Overall, the comparison highlights that the IB3 condition promotes improved toughness, whereas IB2 reflects more brittle failure characteristics.

5.2. Implications on the fracture of granular materials and rocks

The results also have broader implications for the understanding of the fracture behavior of granular materials and rocks. At lower mode mixities, where loading is dominated by opening stresses (Mode I), fracture resistance is very low, meaning that cracks can propagate easily along grain boundaries or weak mineral interfaces. As mode II shear dominated regimes evolve, this behavior suggests that granular microstructures dissipate more energy under shear through mechanisms such as crack deflection, grain interlocking, boundary sliding, and frictional resistance. These processes hinder crack growth and effectively toughen the material.

At high mode mixities, fracture resistance tends to increase, indicating a saturation point where additional shear significantly increases toughness. These findings highlight those granular rocks are less tough under pure tensile stresses, and much tougher under combined tensile-shear conditions. This has implications for predicting natural fracture processes, slope stability, fault mechanics, and engineering applications where granular materials are subjected to complex loading paths,

showing that fracture resistance is not constant but evolves strongly with stress mode.

Comparisons between experimental results and the simplified zone model offer valuable insights into the fracture behavior of granular materials and rocks. The results show that while the simplified zone model can approximate fracture resistance under Near-Mode-I conditions or in some material sets (e.g., IB2 Set B and IB3 Set B), it fails to capture the progressive increase in energy release rate observed experimentally at higher mode mixities in other sets (e.g., IB2 Set A and IB3 Set A).

The implication for granular materials and rocks is that their fracture resistance is not fixed, but strongly dependent on the stress mode and the details of their microstructure. The results suggest that the resistance curve behavior is associated with a crack tip shielding mechanisms and ligament bridging. Under crack opening dominated conditions, cracks exploit weak cleavage joints, thus making it to shield cracks at lower energy release rate. However, as the proportion of shear for mode II loading is increased, the roughness administered in the new interface results in increasing energy release rate with increasing crack extension. As the proportion of shear or mode II loading is increased, the roughness administered in the interfaces results in increasing resistance curve behavior and results always not fully captured by the model.

The current work suggests that the linear-elastic models may underestimate the overall resistance curve behaviour, especially at the highest mode mixity level. Hence, the drilling conditions that are associated with some shear dominated conditions may be more severe than those suggested by some of the predictions especially at high mode mixities. In any case, the current work provides an approach for testing fracture resistance under loading conditions between pure mode I and near pure mode II. Further work is needed to relate the drilling stress state to the resistance curve that have been presented here for barite rocks. These are clearly some of the challenges for future work.

5.3. Conclusions

1. The trends in the fracture resistance of barite rocks can be predicted by the zone model over a range of mode mixities between pure mode

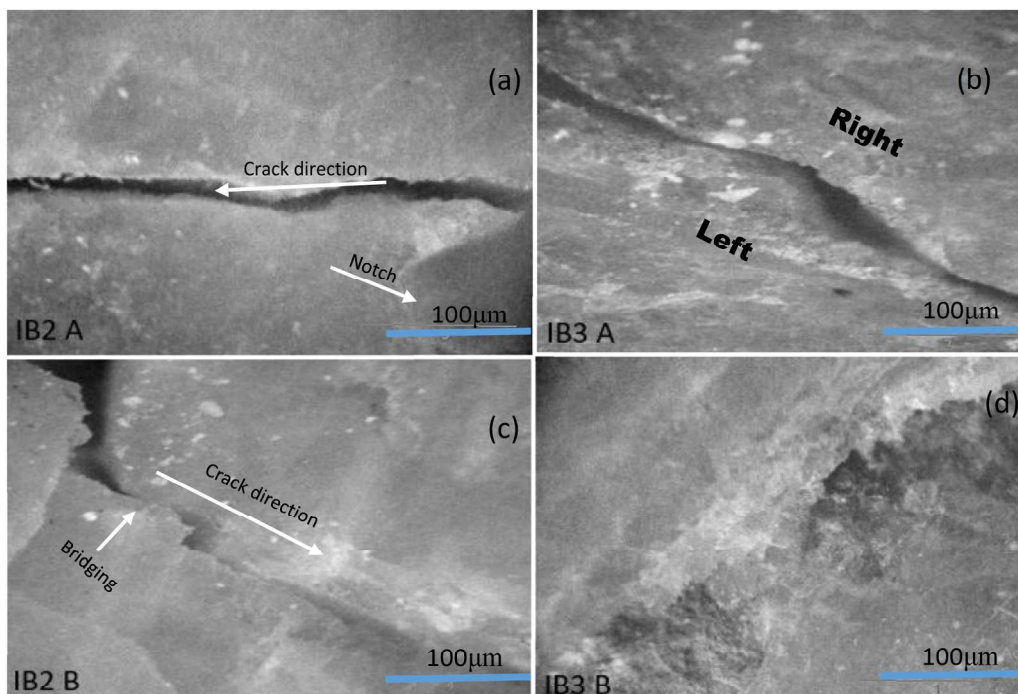


Fig. 7. Cross-sectional optical images of fractured interfaces of (a) IB2 A (b) IB3 A (c) IB2 B and (d) IB3 B.

I and pure mode II. The observed fracture resistance is associated with toughening due to increased shear and crack-tip shielding due to crack bridging and crack deflection.

- At low mode mixities, barite rocks exhibit low fracture resistance due to easy crack propagation along weak cleavage planes or smooth mineral grains that have very low initiation toughness values. However, at high mode mixities, fracture resistance is enhanced as cracks are faced with tough, mismatched interfaces, where microstructural toughening processes, involving crack deflection, and grain boundary sliding, come into play.
- Crack paths are very dependent on mode mixity. At low mode mixities, cracks tend to pass through fairly smooth paths along weak cleavage planes. However, under mixed-mode or shear-dominated conditions, crack deflection is more likely to occur, along with crack bridging, which result in increased toughening and enhanced resistance-curve behavior.
- The insights from the current study can be used to guide the design of drilling operations in which barite is used under mixed-mode loading conditions. The current work suggests that fracture initiation and crack growth resistance can be modeled using resistance curves and interfacial fracture mechanics models over a range of mode mixities. The underlying crack paths and toughening mechanism also depend on mode mixity and the microstructure of barite.

CRedit authorship contribution statement

H.A. Adamu: Writing – review & editing, Writing – original draft, Methodology, Investigation, Data curation, Conceptualization. **D.O. Afolayan:** Writing – review & editing, Supervision, Resources, Methodology, Funding acquisition, Data curation, Conceptualization. **O.S. O. Ogunmodimu:** Writing – review & editing, Supervision, Resources, Methodology, Formal analysis. **S.I. Olajuyi:** Writing – review & editing, Writing – original draft, Methodology. **N.Y. Dzade:** Writing – review & editing, Supervision, Resources, Investigation, Conceptualization. **W.O. Soboyejo:** Writing – review & editing, Visualization, Supervision, Resources, Methodology, Funding acquisition, Formal analysis, Conceptualization.

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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