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# 海/陆相页岩微观力学性质压痕测试研究

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**摘要:**页岩微观力学性质对页岩油气储层钻井、完井、压裂改造具有关键影响。利用X射线衍射、微观结构扫描电镜及压痕测试等手段对四川盆地海相页岩和内蒙古石拐盆地陆相页岩进行研究,结果表明:海相页岩由石英、方解石、白云石及黄铁矿等自生矿物组成,微观结构较为致密,维氏硬度平均值为2.43 kN/mm<sup>2</sup>,岩石坚硬、刚度大,储层破裂压力较高;陆相页岩由石英、钠长石、高岭石和蒙脱石等外源矿物组成,黏土矿物含量高是其显著特征,粒间孔隙发育,结构相对疏松,其维氏硬度平均值为0.80 kN/mm<sup>2</sup>,储层破裂压力较低。海相页岩不同方向上压痕位移差异大,易形成辐射裂缝(纹),其脆性强,储层压裂可采取低裂缝净压力以减小裂缝后期回弹;而陆相页岩不同方向上压痕位移相似且不易形成辐射裂缝,其脆性弱,储层压裂可考虑高裂缝净压力加载,以保障压裂裂缝残余宽度。

**关键词:**海/陆相页岩;微观结构;维氏硬度;压痕实验;页岩储层压裂;裂缝回弹效应

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## Micromechanical characteristics of marine/continental shale based on indentation test

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**Abstract:** Micromechanical properties of the shale play a key role in the drilling, completion, and fracturing of reservoirs. In this paper, the marine shale in Sichuan Basin and continental shale in Shiguai Basin of Inner Mongolia were studied by XRD, SEM, and indentation test. The results show that marine shale in Sichuan Basin is mainly composed of authigenic minerals such as quartz, calcite, dolomite, and pyrite, while continental shale is composed of exogenous minerals such as quartz, kaolinite, montmorillonite, and albite. The microscopic structure of marine shale is relatively compact, while the continental shale develops intergranular pores and the structure is relatively loose. The indentation test results show that the average Vickers hardness ( $H_v$ ) of marine shale is above 2.43 kN/mm<sup>2</sup>; the rock stiffness is hard and stiff, and the breakdown pressure required for the reservoir is high. The average  $H_v$  of continental shale is 0.80 kN/mm<sup>2</sup>; the rock is relatively weak, and the breakdown pressure of the reservoir is low. Marine shale has a large indentation displacement difference in different directions and is prone to radiation fractures, indicating that the rock brittleness is significant. Continental shale has similar indentation displacement in different directions and is not prone to radiation fractures, indicating that the rock brittleness is relatively weak. Combined with the energy theory, low net fracture pressure is adopted in marine shale reservoir fracturing to reduce the fracture rebound in the later period, while high net fracture pressure can be considered in continental shale reservoir fracturing to ensure fracture residual width.

**Key words:** marine/continental shale; microstructure; Vickers hardness; indentation test; shale reservoir fracturing; frac-

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ture rebound effect

陆相页岩油气开发对于保障能源安全、实现油气自主供给具有战略意义<sup>[1-3]</sup>。长期以来,围绕四川盆地海相页岩岩石力学性质方面的研究较多,针对页岩岩石物性与力学性能<sup>[4-7]</sup>、各向异性<sup>[8-9]</sup>、对井壁稳定性影响<sup>[10-11]</sup>、岩石压裂力学性质及造缝效果<sup>[12-15]</sup>等方面开展了大量研究,为海相页岩气开发提供有力支撑。近年来,由于霍普金森压杆等高速冲击加载实验手段和分析理论的交叉,页岩动态断裂力学及断裂加载率效应方面研究<sup>[16-18]</sup>备受关注,但相关研究在压裂中的尺寸效应问题有待深入。有必要提及的是,海/陆相页岩在矿物组成、岩石物性、岩石力学性质等方面差异显著<sup>[19-22]</sup>。随着中国陆相页岩油气开发的推进,目前针对陆相页岩储层岩石力学性质、储层压裂裂缝物理模拟方面的研究工作进展较快,但研究深度与海相页岩相比仍需加强,亟待开展海/陆相页岩在岩石力学、岩石断裂力学等方面的差异性分析,从而为陆相页岩在油气钻井、完井、储层压裂改造等方面提供科学依据<sup>[23-24]</sup>。由于页岩比煤岩、砂岩更致密,宏观尺度岩石表征已不能满足致密页岩油气挖掘的需求,因此摸清页岩微观力学性质,对构建页岩储层地质力学模型、提升页岩储层钻完井效率及评价页岩储层可压性等具有重要意义。前人针对海相页岩利用纳米压痕及压痕法已开展了大量研究工作<sup>[25-28]</sup>,然而针对陆相页岩尤其与海相页岩微观力学性质对比分析方面的研究仍较少。

为此,利用常规压痕实验对四川盆地龙马溪组海相页岩和内蒙古石拐盆地厂汉沟组陆相页岩微观力学性质进行实验研究,以获取海/陆相页岩维氏硬度数据和压痕形态特征图像,分析不同方向上压痕位移随载荷的变化规律,并构建了一套基于压痕

位移比的页岩脆性评价方法,结合弹性能量理论,分析海/陆相页岩储层压裂裂缝的回弹效应差异对压裂裂缝残留宽度的影响,并提出海/陆相页岩储层针对性压裂建议,以期陆相页岩油气开发储层压裂改造策略优化、页岩储层“个性化”压裂提供参考。

## 1 样品特征与实验方法

### 1.1 海/陆相页岩宏观特征

海相页岩采自四川盆地龙马溪组,黑色有机质含量高,结构致密,断口呈眼球状、贝壳状,其脆性显著;陆相页岩采自内蒙古石拐盆地厂汉沟组,为陆相湖盆环境沉积,呈深灰、黑灰色,有机质含量相对较低,结构相对疏松,指甲可刻划,层理结构清晰。从沉积成岩环境、水动力条件来看,海相页岩水动力较弱,矿物颗粒间结合强度较高;陆相页岩水动力较强且成岩矿物间以机械压实作用为主,矿物颗粒间结合强度明显弱于海相页岩。

### 1.2 海/陆相页岩矿物组成特征

X射线衍射和微观结构扫描电镜显示:海相页岩主要由石英、方解石、白云石和黄铁矿等矿物组成(图1a,图2a—2c),自生矿物比例较高,具有典型海相沉积成岩特征<sup>[21]</sup>;陆相页岩主要由石英、钠长石、高岭石和蒙脱石等组成(图1b,图2d—2f),均为外源矿物后期搬运沉积成岩。依据混合律理论<sup>[29]</sup>,海相页岩主要由脆性矿物组成,而陆相页岩含大量黏土矿物,因而海相页岩脆性较强。

### 1.3 海/陆相页岩微观结构特征

由图2所示,整体上四川盆地海相页岩结构较为致密,发育大量脆性断口。而内蒙古石拐盆地陆

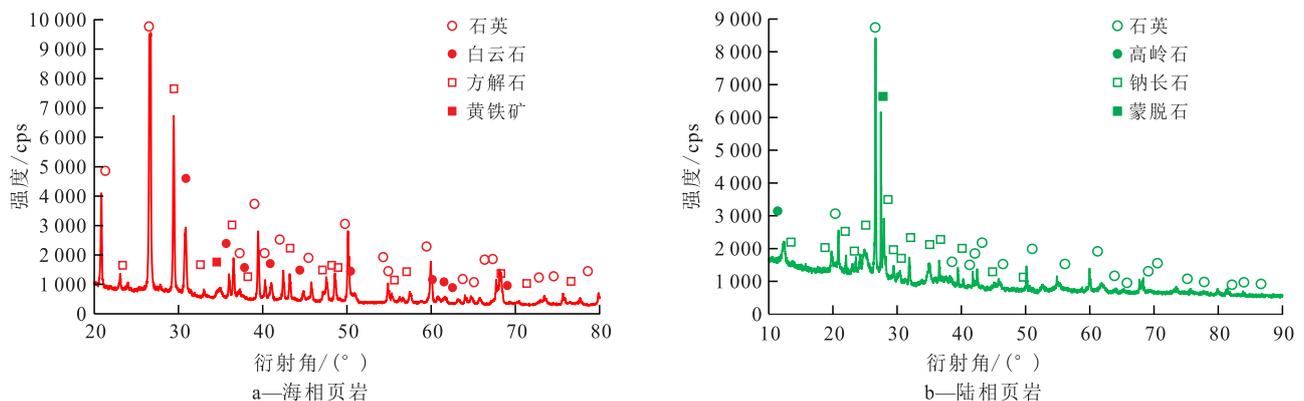


图1 海/陆相页岩矿物特征X射线衍射分析结果

Fig.1 XRD analysis results of mineral characteristics of marine/continental shales

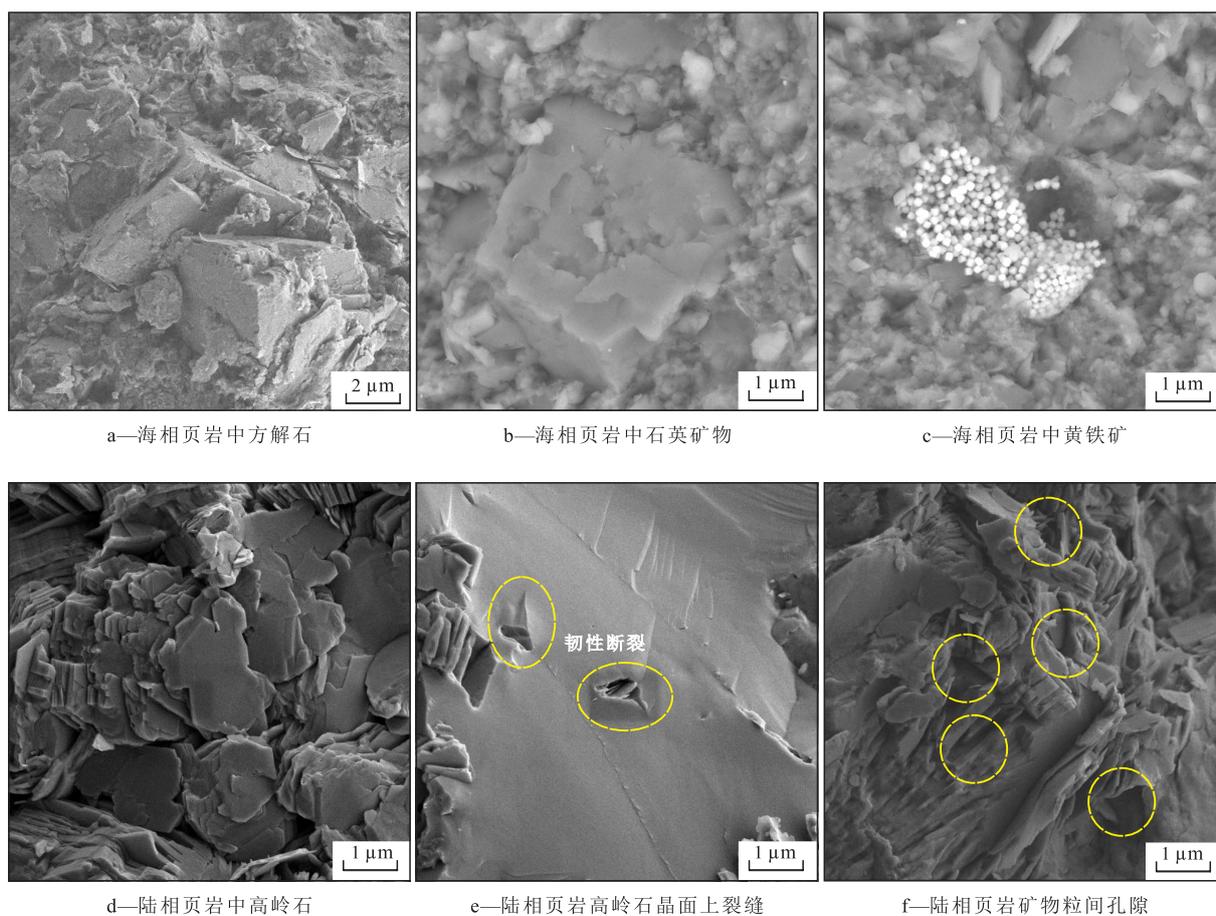


图2 海/陆相页岩微观结构特征SEM照片

Fig.2 SEM images of microstructure characteristics of marine/continental shales

相页岩粒(晶)间孔隙非常发育,岩石结构较为疏松。值得注意的是,扫描电镜照片显示陆相页岩中高岭石晶体界面上压痕为韧性断裂形式(表现为裂缝附近无辐射破坏等现象),也指示陆相页岩的力学行为偏塑性。结合矿物组成和微观结构特征分析,认为相对四川盆地海相页岩,内蒙古石拐盆地陆相页岩成岩矿物以高岭石等黏土矿物(图2d—2f)为主,压裂中水基压裂液进入储层会与上述矿物反应形成水敏性伤害<sup>[22]</sup>,需引起重视。

#### 1.4 压痕测试实验方法

压痕测试的具体实验步骤包括:①将海/陆相岩样切割为1.5 cm×1.5 cm×1.5 cm的立方体,样品顶面抛光,且顶、底面保持平行。②利用显微硬度计(图3)对海/陆相岩样进行压痕测试,硬度计加载时压头接触样品时间为15 s,其中海相岩样压头施加载荷( $P$ )设定为1,2,3,5和10 N共5档,考虑陆相页岩相对较软,压头施加载荷仅设定为1,2和3 N共3档。③显微镜下测量不同压头施加载荷下岩样顶面 $X, Y$ 两个方向的压痕位移。依据维氏硬度公式计算岩样硬度,表达式为<sup>[29]</sup>:

$$H_v = 1.854 \frac{P}{(XY)^2} \quad (1)$$



图3 压痕测试实验装置

Fig.3 Layout of indentation test

## 2 结果与分析

### 2.1 页岩维氏硬度特征

本次实验得到四川盆地海相页岩和内蒙古石

拐盆地陆相页岩维氏硬度平均值分别为 2.43 和 0.80 kN/mm<sup>2</sup>,前者是后者的 3~4 倍,说明海相页岩的致密度远比陆相高,抵抗外载荷侵入能量更强,岩石破碎需要消耗更多能量。

值得指出的是,海相页岩随载荷增加,维氏硬度值离散性加强;而陆相页岩随载荷增加,维氏硬度值的离散性则没有明显变化(图4),这也反映出海相页岩材料的脆性相对较强。

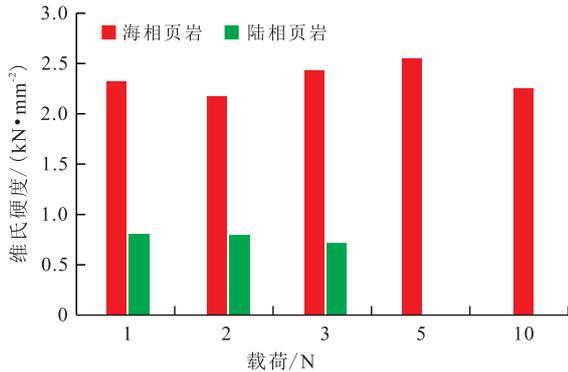


图4 海/陆相页岩维氏硬度特征

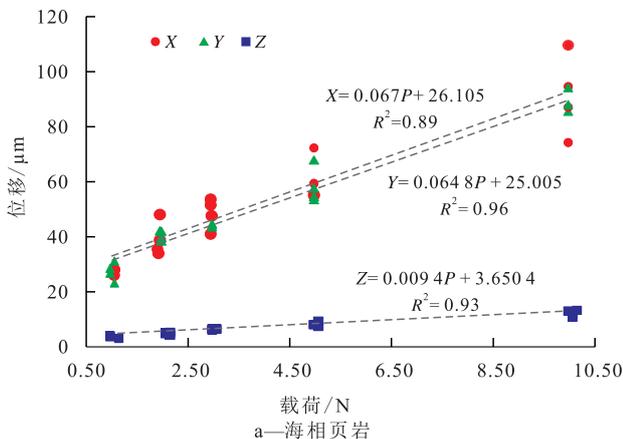
Fig.4 Vickers hardness characteristics of marine/continental shales

### 2.2 压痕位移随载荷的变化规律

对海/陆相页岩岩样在 X, Y 和 Z 方向上形成的压痕位移与载荷之间的关系(图5)进行拟合分析,认为随着压头载荷增大,在海/陆相岩样 X, Y 和 Z 方向上形成的压痕位移均呈线性增长,说明本次实验所用的岩样基质具有较好的均质性。随着载荷增加,海相页岩压痕位移的离散性增强(图5a),这与海相岩石脆性较强的判断相吻合;随载荷增大,陆相页岩压痕位移的离散性并未增强(图5b),表明陆相页岩力学性质偏塑性。

### 2.3 海/陆相页岩压痕形态特征

利用显微镜捕捉了海/陆相页岩压痕形态特征



(图6)。镜下照片显示,随着压头载荷增大,页岩岩样在 X, Y 方向上的压痕位移均增加。其中海相页岩岩样在压头侵入下形成不规则压痕形态, X, Y 方向上的压痕位移差异较大,且压痕端部多形成辐射状裂纹,呈较为明显的脆性断裂。陆相页岩岩样压痕呈规则菱形深凹坑形态, X, Y 方向上的压痕位移随载荷增加而快速增大,表明陆相页岩岩样偏软,抵抗外界侵入能力较弱,压痕四周边界清晰、形态规整;值得注意的是陆相页岩岩样表面压痕端部少有辐射裂纹,表明陆相页岩岩样在外载作用下呈塑性力学特性,对于储层压裂改造不利<sup>[23-24]</sup>。

## 3 海/陆相页岩微观力学性能对储层压裂造缝的启示

### 3.1 页岩脆性特征的评价

通常维氏硬度的离散性随载荷的变化规律与测试对象材料脆性的强弱有关。为此,将四川盆地海相页岩、内蒙古石拐盆地陆相页岩以及内蒙古二道岭矿区无烟煤压痕测试结果进行比较,分析岩石维氏硬度随压头载荷增大的变化规律,并对3类岩样维氏硬度的离散性进行比较(图7a),发现随着载荷增大,海相页岩维氏硬度的离散性显著高于陆相页岩和无烟煤,这与3类岩石宏观力学性质和断裂特征吻合。

依据 X, Y 方向上的压痕位移比提出一种页岩脆性评价方法,当压痕位移比大于 0.1 时,岩石为脆性;反之为塑性。计算可知海相页岩压痕位移比普遍大于 0.1, 岩石脆性较强,而与压头载荷无明显相关性(图7b),说明脆性为岩石固有属性,与外部加载条件关系不大;而陆相页岩压痕位移比小于 0.1, 且随载荷增加,压痕位移比具有减小的趋势。

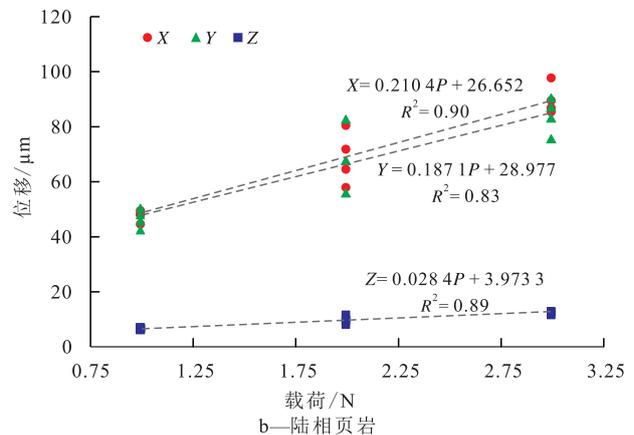


图5 海/陆相页岩压痕位移随压头载荷的变化规律

Fig.5 Indentation displacement of marine/continental shales varies with indenter load

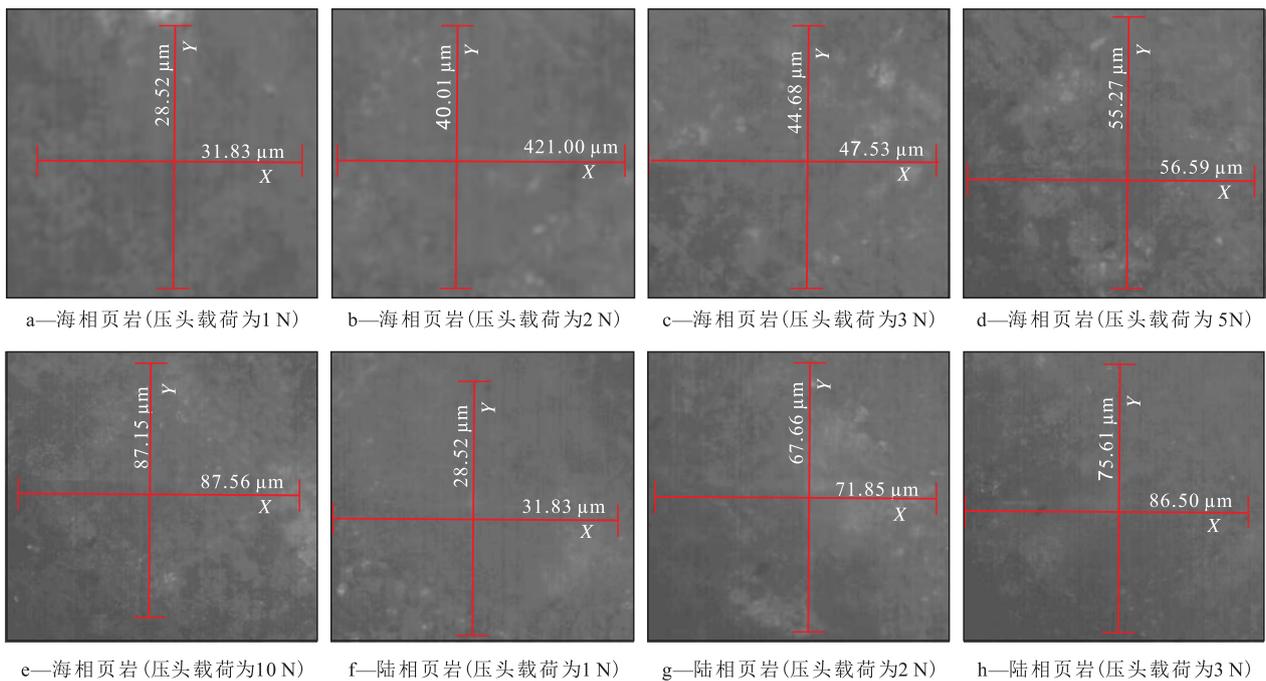


图6 海/陆相页岩压痕形态特征

Fig.6 Indentation characteristics of marine/continental shales

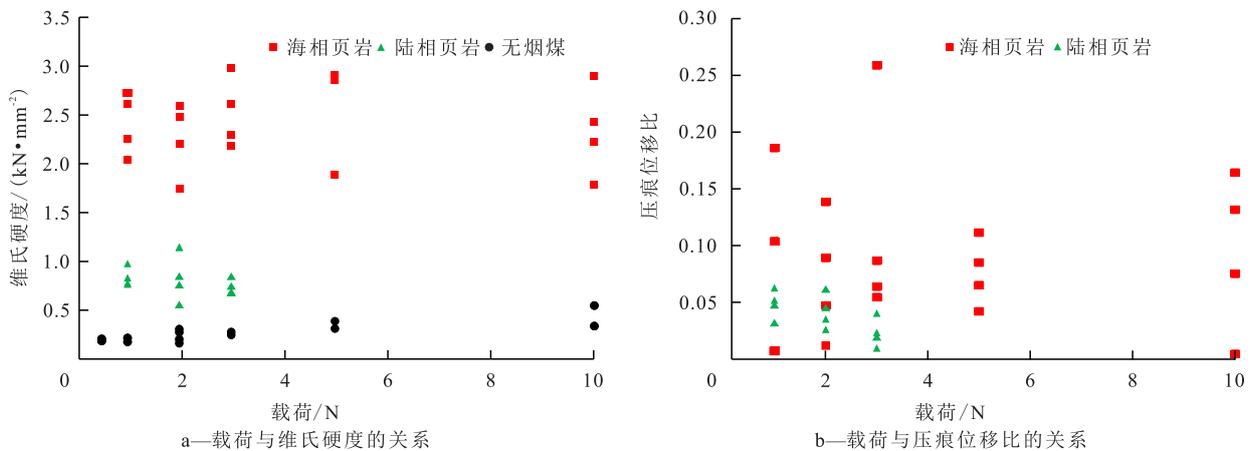


图7 海/陆相页岩维氏硬度与压痕位移比随载荷的变化规律

Fig.7 Vickers hardness and indentation displacement ratio of marine/continental shales varies with indenter load

### 3.2 压缩能量对储层压裂的启示

当显微硬度计压头压入岩样内部并到达最大侵入深度时,岩样在X,Y和Z方向上的压痕位移均达到最大值。当压头从岩样撤出后,由于岩石弹性效应压痕空间将回缩,其中在X,Y方向上压痕半径和在Z方向上压痕深度分别缩减为残留压痕半径( $C_r$ )和残留压痕深度( $Z_r$ )(图8)。依据材料力学原理,当测试材料为脆性时,压痕空间回弹程度高,即 $C_r/C_{max}$ 或 $Z_r/Z_{max}$ 趋近于0;而材料为塑性材料弹性模量小、刚度低,压痕空间回弹程度低,即 $C_r/C_{max}$ 或 $Z_r/Z_{max}$ 趋近于1。加载曲线和卸载曲线及横轴所包围面积( $S_1$ )为页岩材料变形所吸收的能量;从卸载曲

线最高点做垂线,该垂线与卸载曲线及位移轴所包围面积( $S_2$ )即为弹性形变所需能量,是可恢复的<sup>[30-32]</sup>。

依据本次压痕实验结果和脆性程度,提出四川盆地海相页岩和内蒙古石拐盆地陆相页岩的压缩能量模型,对于脆性显著的海相页岩,为增大 $C_r/C_{max}$ ,形成有效压裂裂缝空间,需采取相对较低的裂缝净压力进行加载,从而减小弹性形变所需能量,保持塑性变形所吸收的能量,规避压裂裂缝回弹(图8a);对于脆性较弱的陆相页岩,为增大 $C_r/C_{max}$ ,减小裂缝回弹,应以较大的裂缝净压力加载(图8b)。

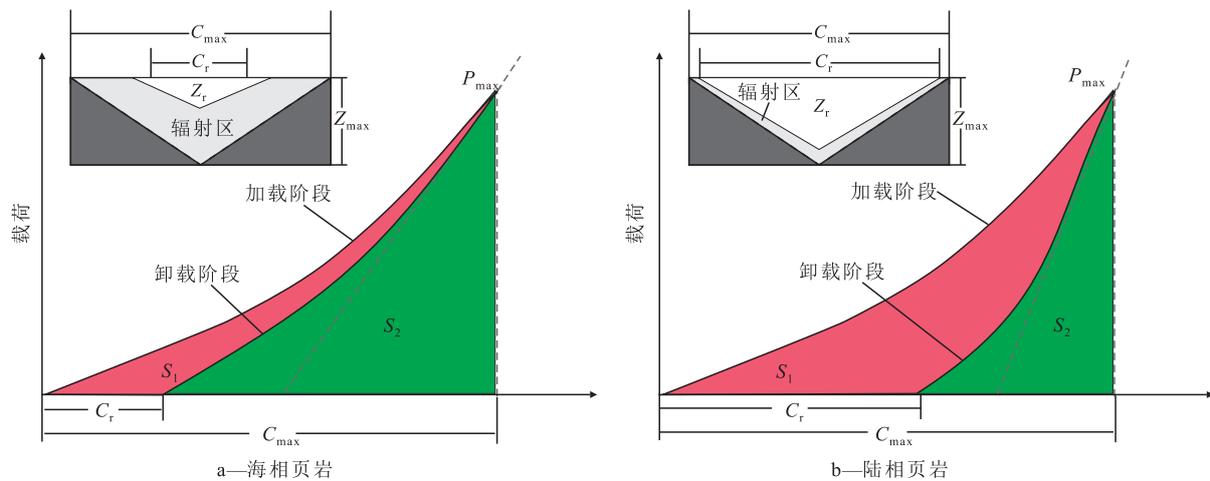


图8 海/陆相页岩压缩能量模式

Fig.8 Compressional energy modes of marine/continental shales

## 4 结论

四川盆地海相页岩维氏硬度平均值为2.43 kN/mm<sup>2</sup>,岩石刚度大,储层破裂压力较高;内蒙古石拐盆地陆相页岩维氏硬度平均值为0.80 kN/mm<sup>2</sup>,岩石刚度相对较低,储层破裂压力较低。四川盆地海相页岩由石英、方解石、白云石及黄铁矿等自生矿物组成,陆相页岩则由石英、钠长石及高岭石等外源矿物组成,黏土矿物含量高是其显著特征,也是导致陆相页岩刚度较低的重要原因;海相页岩微观结构较为致密,而陆相页岩发育矿物粒间孔隙,结构相对疏松,是导致陆相页岩硬度较低的内在因素。海相页岩在X,Y和Z方向的压痕位移差异较大且易形成辐射裂缝(纹),表明岩石脆性显著;而陆相页岩在各方向上的压痕位移相近且不易形成辐射裂缝,指示岩石脆性相对较弱。结合能量理论提出海相页岩储层压裂时采取低裂缝净压力减小裂缝后期回弹,而陆相页岩储层压裂时可考虑高裂缝净压力加载,以保障压裂裂缝的残余宽度。

### 符号解释

- C——压痕半径,μm;
- C<sub>max</sub>——最大压痕半径,μm;
- C<sub>r</sub>——残留压痕半径,μm;
- H<sub>v</sub>——维氏硬度,kN/mm<sup>2</sup>;
- P——压头载荷,N;
- P<sub>max</sub>——最大压头载荷,N;
- S<sub>1</sub>——加载曲线和卸载曲线及横轴包围面积,μm<sup>2</sup>;
- S<sub>2</sub>——从卸载曲线最高点做垂线,该垂线与卸载曲线及

- 位移轴包围面积,μm<sup>2</sup>;
- X,Y——不同方向上的压痕半径,μm;
- Z——压痕深度,μm;
- Z<sub>max</sub>——最大压痕深度,μm;
- Z<sub>r</sub>——残留压痕深度,μm。

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