

氧化液作用下富有机质页岩裂缝应力敏感性

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摘要:页岩中有机质和黄铁矿等还原环境沉积产物易被氧化,氧化溶蚀作用虽有利于改造页岩基质渗流通道,但对裂缝渗透率应力敏感性的影响尚不明确。将川东龙马溪组露头页岩裂缝岩样分别浸泡在蒸馏水和质量分数为15%的氧化液中,分别静置24,48和72 h,取出烘干后测定其渗透率,将浸泡72 h的岩样进行应力敏感性评价及离子组成分析,利用X衍射和扫描电镜分析浸泡前、后页岩组分及结构变化。渗透率测定和应力敏感性评价结果表明:经蒸馏水浸泡的岩样渗透率提高0.42~6.15倍,应力敏感系数为0.514~0.587;经氧化液浸泡的岩样渗透率提高5.53~65.45倍,应力敏感系数为0.482~0.517。离子组成分析和X衍射测试结果显示,白云石和方解石含量大幅减小,黄铁矿甚至完全消失,而粘土矿物、石英和长石含量稳定,说明岩样裂缝面发生了选择性氧化溶蚀。岩样断面观察和扫描电镜图像揭示,氧化液沿页理和裂缝渗入,诱发裂缝萌生、扩展,改善了微纳米级孔隙,证明氧化溶蚀作用促使页岩孔隙溶扩。裂缝面矿物的选择性氧化溶蚀及其诱发的孔隙溶扩,使裂缝闭合过程中仍能保持相对较高的渗流能力,降低了应力敏感损害程度。

关键词:页岩气层 天然裂缝 应力敏感性 氧化液 氧化溶蚀 孔隙溶扩

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Fracture stress sensitivity of organic-rich shale under the action of oxidation fluid

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Abstract: Organic matter and pyrite in shale deposited in reduction environment, which are easy to be oxidized and dissolved. Oxidation is useful to the reform of the seepage channels of shale matrix, but its influence on the stress sensitivity of permeability of shale fracture is not clear. Artificial fractured samples were imbibed in distilled water or 15% oxidation liquid for 24, 48, and 72 h respectively. Samples permeability were measured after the samples were dried. The stress sensitivity of the samples that soaked for 72 h were evaluated. Meanwhile, the ion type and concentration of the solution were tested, and the change of shale composition and structure before and after oxidation were characterized by X-ray diffraction (XRD) and scan electron microscope (SEM). Results show that the permeability of the samples soaked in distilled water is increased by 0.42–6.15 times and the stress sensitivity coefficients range from 0.514 to 0.587. The permeability of rock samples soaked in the oxidation fluid is increased by 5.53–65.45 times, while the stress sensitivity coefficients range from 0.482 to 0.517. Ion analysis and XRD test results show that the oxidation can significantly reduce the content of dolomite and calcite, and pyrite even disappears completely. But the content of clay mineral, quartz and feldspar remains unchanged. It means that selective oxidative dissolution occurs on the fracture plane. The section observation and SEM image of the sample confirm that oxidation fluid infiltrates along the crack and the lamellation, inducing and expanding cracks and improving micro- and nano porosity. It is proved that the oxidation results in the expansion of pore and crack in shale. Selec-

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tive oxidative dissolution of minerals on the fracture plane and the consequent expansion of pore and crack caused by oxidation, which helps to remain relatively high flow capacity during fracture closure and reduce the stress sensitivity damage.

Key words: shale gas reservoirs; natural fracture; stress sensitivity; oxidation fluid; oxidative dissolution; expansion of pore and crack

富有机质页岩氧化对提高页岩气藏采收率具有重要意义^[1-2]。页岩含有机质、黄铁矿等还原环境沉积产物^[3],易在富氧条件下发生氧化溶蚀,形成溶蚀孔缝,从而提升页岩孔缝系统的传输能力^[4-5]。巫锡勇等研究发现,黑色页岩在地表环境下极易氧化和风化,从而破坏岩层结构,改变岩体力学性质^[6]。页岩层理和天然裂缝发育,水力压裂使天然裂缝网络开启,构成了复杂的裂缝网络^[7]。在工作液长期浸润下,页岩物理化学性质发生改变,使储层的应力敏感性变得更复杂^[8]。考虑工作液侵入的页岩气层应力敏感性,有助于认识裂缝的动态变化行为,可以有效地保护天然裂缝,防止人工裂缝闭合,从而保障基质孔喉、天然裂缝和人工裂缝三者之间的多尺度传质效率^[9-11]。

页岩应力敏感性与其矿物组分、孔隙结构密切相关。孟英峰研究发现,页岩吸水导致粘土矿物水化膨胀,水化附加应力改变了原地应力场,造成应力敏感损害^[12]。张浩等认为,人工裂缝、天然裂缝和基质的应力敏感性依次减弱^[13]。游利军等发现裂缝的应力敏感性大于基质,应注重保护裂缝^[14];张睿等通过对页岩矿物组分、力学性质和微观孔隙结构表征,分析了影响应力敏感性的主要因素^[15]。张杜杰等基于对致密砂岩裂缝面在有效应力加载前、后的微观结构表征,发现对于不同宽度的裂缝,裂缝壁面出砂对应力敏感性的影响存在差异^[16]。何金钢等认为,外来流体不配伍诱发的微粒运移、粘土矿物微结构破坏等也会影响应力敏感性^[17]。ZHOU等研究指出,页岩自吸诱发水-岩作用,会削弱裂缝面强度,增强裂缝应力敏感性^[18]。考虑压裂液浸润会增强页岩裂缝应力敏感性^[7],如果氧化液进入页岩气藏浸润页岩,氧化溶蚀产生孔缝提高渗

透率的同时,是否影响裂缝渗透率应力敏感性尚不明确。为此,以川东龙马溪组露头页岩为研究对象,开展氧化液作用下页岩裂缝岩样的应力敏感性评价实验,对比分析氧化对页岩裂缝应力敏感性的影响。结合溶液离子组成分析,利用X衍射和扫描电镜等手段,从页岩组分、结构变化的角度,揭示氧化作用对页岩裂缝应力敏感性的影响机理,以期页岩氧化改造提高气体传输能力的可行性提供技术支撑。

1 实验样品与方法

1.1 实验样品

川东龙马溪组露头页岩矿物组成分析结果表明,该页岩中石英含量为53.5%,长石含量为7.6%,粘土矿物含量为31.9%,其中粘土矿物以伊利石和伊/蒙混层为主,伊利石含量为46.0%,伊/蒙混层含量为42.7%,高岭石含量为11.3%,不含绿泥石。易氧化组分黄铁矿含量为2.6%,有机质含量为3.8%。其中黄铁矿和粘土矿物晶间孔、有机质孔、微裂缝发育(图1)。页岩裂缝岩样基本参数如表1所示。

实验流体为蒸馏水和氧化液。氧化液由蒸馏水和质量分数为15%的氧化剂FDO-1配制而成。

1.2 实验方法

选取川东龙马溪组露头页岩,在同一岩块相近位置沿层理钻取实验所需岩样,岩样经人工造缝后开展应力敏感性评价实验。岩样中人工裂缝为平行于轴线的单条裂缝。LY-1和LY-2岩样用蒸馏水浸泡,LY-3和LY-4岩样用氧化液浸泡。具体实验步骤为:①将岩样分别浸泡在氧化液、蒸馏水中,分别静置24,48和72 h后取出,进行端面拍照。②用

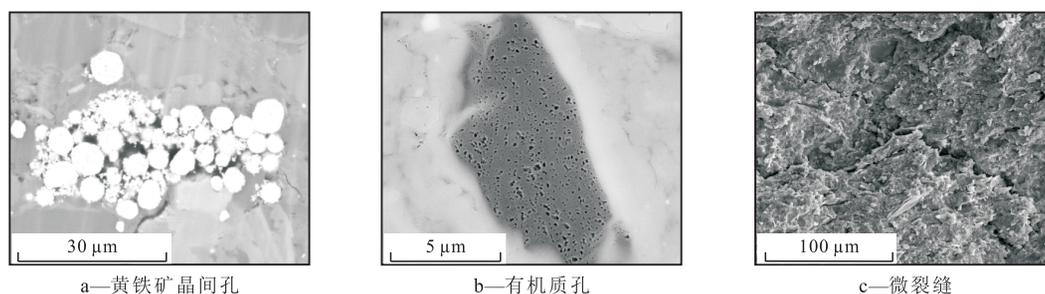


图1 川东龙马溪组露头页岩孔缝特征

Fig.1 Characteristics of pore and crack of outcrop shale samples in the Longmaxi Formation, eastern Sichuan

表1 川东龙马溪组露头页岩岩样基本参数
Table1 Basic parameters of fractured outcrop shale samples in the Longmaxi Formation, eastern Sichuan

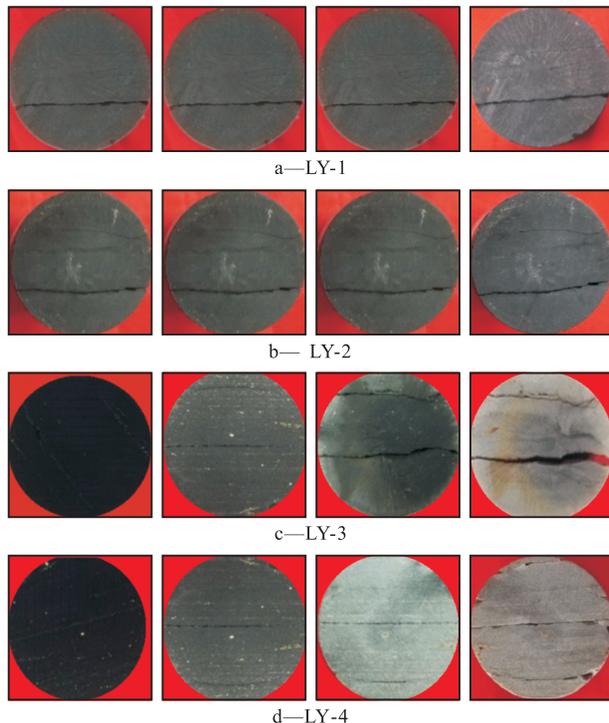
编号	长度/mm	直径/mm	渗透率/mD
LY-1	45.09	24.89	1.76
LY-2	42.11	25.05	0.33
LY-3	34.33	24.94	2.80
LY-4	38.87	25.23	0.31

滚子加热炉在 60 °C 下对岩样恒温干燥 48 h 后,测定其渗透率。③利用 SCMS-II 型高温高压岩心多参数测量系统,对浸泡 72 h 后的岩样分别测定有效应力为 3, 5, 7, 10, 15, 20 和 25 MPa 条件下的渗透率。④对浸泡后的溶液进行离子组成分析,并对浸泡后的岩样进行 X 衍射和扫描电镜分析。采用应力敏感性系数法评价各岩样的应力敏感性^[19-20]。

2 页岩裂缝应力敏感性评价结果

由图2可以看出:LY-1 和 LY-2 岩样在被蒸馏水浸泡过程中,端面形态没有发生明显变化;而经氧化液浸泡后,LY-3 和 LY-4 岩样由灰褐色变为灰白色,且均出现裂缝扩展。

浸泡不同时间后岩样渗透率变化结果(图3)表明,经蒸馏水浸泡 72 h 后,岩样渗透率提高 0.42~



注: 图片由左到右浸泡时间依次为0, 24, 48和72 h

图2 岩样经蒸馏水或氧化液浸泡不同时间后的截面

Fig.2 Cross section of fractured samples after immersion in distilled water or oxidization fluid for different time lengths

6.15 倍;而经氧化液浸泡 72 h 后,岩样渗透率提高显著,为 5.53~65.45 倍。4 块岩样采用相同方式进行人工造缝,LY-4 较 LY-3 岩样渗透率虽低一个数量级,但氧化溶蚀使 LY-4 岩样渗透率提高近 66 倍,且高于 LY-3 岩样氧化后的渗透率。

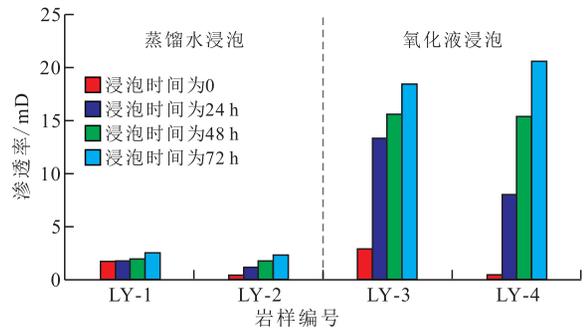


图3 岩样经蒸馏水或氧化液浸泡不同时间后的渗透率

Fig.3 Permeability of fractured samples after immersion in distilled water or oxidization fluid for different time lengths

对于渗透率不同的 LY-1 和 LY-3 岩样,随着有效应力的增加,经蒸馏水和氧化液浸泡岩样的无因次渗透率下降趋势相近,且当有效应力小于 10 MPa 时,二者加载曲线基本重合(图4a);对于渗透率相近的 LY-2 和 LY-4 岩样,经氧化液浸泡的 LY-4 岩样的无因次渗透率下降速率较经蒸馏水浸泡的 LY-2 岩样有所减缓,且相同有效应力下无因次渗透率

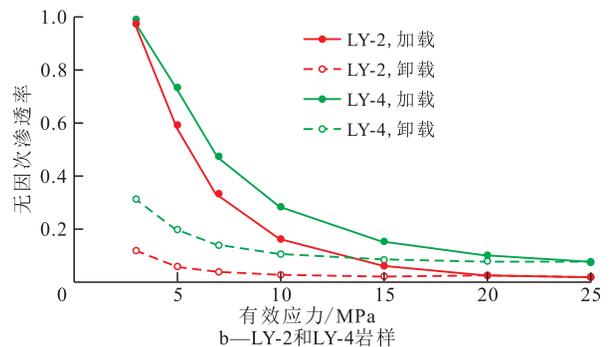
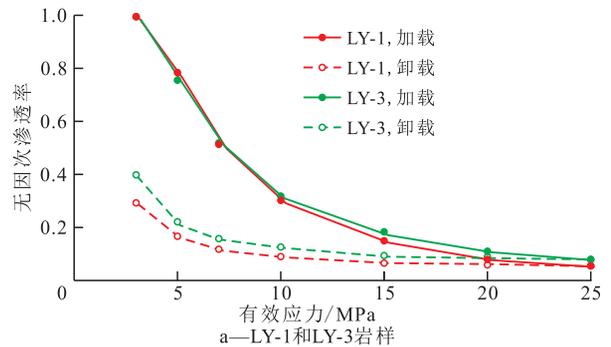


图4 岩样经蒸馏水或氧化液浸泡 72 h 后渗透率随有效应力的变化

Fig.4 Variation of permeability of fractured samples with effective stress after immersion in distilled water or oxidizing fluid for 72 h

也更大(图4b)。4条卸载曲线均明显低于加载曲线,说明微凸体在压缩变形时所存储的应变能大于卸载恢复时所释放的应变能^[21]。对比有效应力卸载曲线发现,经氧化液浸泡的岩样,有效应力卸载后渗透率恢复率更高;尤其对于LY-1和LY-3岩样,加载曲线甚至在有效应力小于10 MPa时重合,而在相同卸载应力下,氧化液浸泡的LY-3岩样均高于蒸馏水浸泡的LY-1岩样,表明氧化作用对应力敏感损害有所缓解。

经蒸馏水浸泡后,LY-1和LY-2岩样的应力敏感系数分别为0.514和0.587,应力敏感性为中等偏强;氧化液浸泡后LY-3和LY-4岩样的应力敏感系数分别为0.517和0.482,应力敏感性分别为中等偏强和中等偏弱,说明富有机质页岩氧化对缓解蒸馏水诱发的页岩裂缝应力敏感损害有积极作用。

3 氧化溶蚀影响页岩裂缝应力敏感性的机理及应用

3.1 页岩裂缝面矿物的选择性氧化溶蚀

岩样经蒸馏水浸泡后,溶液中的阳离子主要包括 Na^+ 、 Mg^{2+} 和 K^+ ,含有微量的 Fe^{3+} 和 Ca^{2+} ,其质量浓度分别仅为0.12和0.06 mg/L。而经氧化液浸泡后, K^+ 和 Na^+ 质量浓度相对稳定, Ca^{2+} 、 Mg^{2+} 和 Fe^{3+} 质量浓度均显著增加,三者反应后与反应前的质量浓度比分别为12 208,36和3 367(图5)。

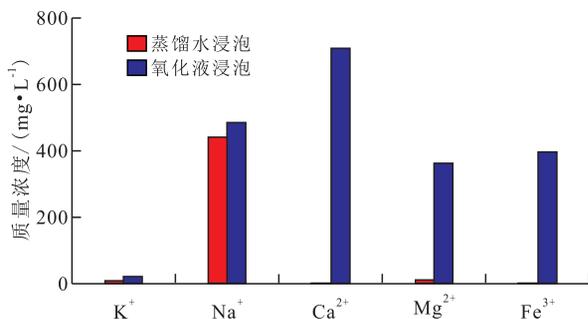


图5 浸泡岩样72 h后的蒸馏水或氧化液中离子浓度

Fig.5 Ion concentration of fractured samples after immersion in distilled water or in oxidization fluid for 72 h

对比经蒸馏水和氧化液浸泡后岩样的X衍射测试结果(图6)发现:经氧化液浸泡后,岩样中的粘土矿物、石英和长石含量无明显的变化,黄铁矿被完全溶蚀;有机质氧化反应产生有机酸^[3],使得方解石和白云石含量大幅减少,样品溶蚀率达86%^[4]。氧化液对龙马溪组富有机质页岩内溶蚀的无机矿物主要有黄铁矿、方解石和白云石,这与岩样经蒸馏水、氧化液浸泡后溶液阳离子组分测试结果一致。

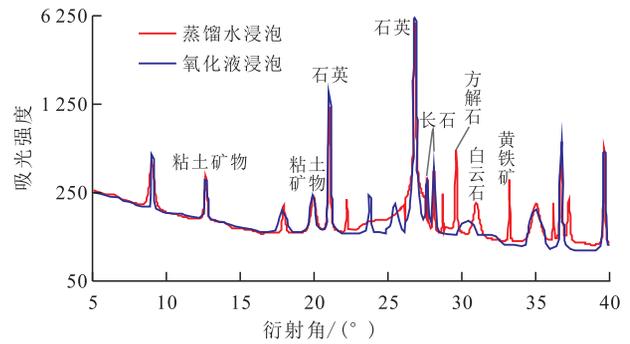


图6 岩样经蒸馏水或氧化液浸泡后X衍射测试结果

Fig.6 Results of the X-ray diffraction of fractured samples after immersion in distilled water or in oxidization fluid

分析认为,氧化液优先沿裂缝面迅速铺展,与黄铁矿、方解石、白云石和有机质接触后反应,进而影响裂缝应力敏感性。这是因为:①氧化溶蚀作用改善了纳米级孔隙,溶蚀孔缝增强了原生孔隙的连通性,提高了页岩渗透率(图3)。②微凸体的塑性变形会显著加剧裂缝的闭合^[22],有效应力加载与卸载曲线不重合,表明蒸馏水和氧化液浸泡后裂缝面微凸体均发生了明显的塑性变形,微凸体内部产生了损伤,能量发生了耗散。相对蒸馏水浸泡后的裂缝,选择性氧化溶蚀提高了裂缝面的导流能力,即使在有效应力加载下促使裂缝闭合过程中,仍能保持一定的渗透率,一定程度上缓解了蒸馏水引发的应力敏感性。

3.2 氧化溶蚀诱发孔缝溶扩效应

水相通过自发渗吸的方式进入页岩内部^[23],易诱发页岩粘土矿物水化膨胀并产生水合力,水合力作用裂缝尖端促进裂缝扩展^[24-25]。页岩经蒸馏水浸泡后,其截面裂缝形态变化不明显;而经氧化液浸泡相同时间后,其截面裂缝扩展特征显著(图2)。笔者认为,氧化溶蚀协同水合力加速了页岩裂缝扩展,大大提高了页岩渗透率(图3),使得在有效应力加载过程中渗透率降幅相对较小,表现为页岩应力敏感性相对减弱。

CHEN等通过实验发现:龙马溪组露头页岩有机质氧化去除率高达87%;压汞法数据显示,未浸泡岩样孔径主要为5~50 nm,对应单位质量的孔隙体积占比为71.62%,而经氧化液浸泡24 h后,其孔径主要为50~500 nm,对应单位质量的孔隙体积占比为68.44%^[4]。对比分别经蒸馏水、氧化液浸泡后页岩的扫描电镜图片也发现,氧化溶蚀作用增大了孔径(图7)。张睿等研究指出,当对页岩渗透率起主要贡献的孔隙孔径主要为10~50 nm时,应力敏感性;而当对渗透率起主要贡献的孔隙孔径大于50

nm时,应力敏感性减弱^[15]。有效应力加载过程中平均毛管半径保留率更高,小孔的比例越低,有效应力加载后提供给大孔的渗流通道更多^[26]。氧化溶蚀诱发页岩纳米级孔隙扩径,因而削弱了页岩的应力敏感性。

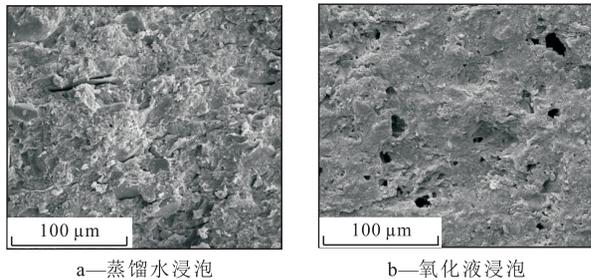


图7 岩样经蒸馏水或氧化液浸泡后扫描电镜结果

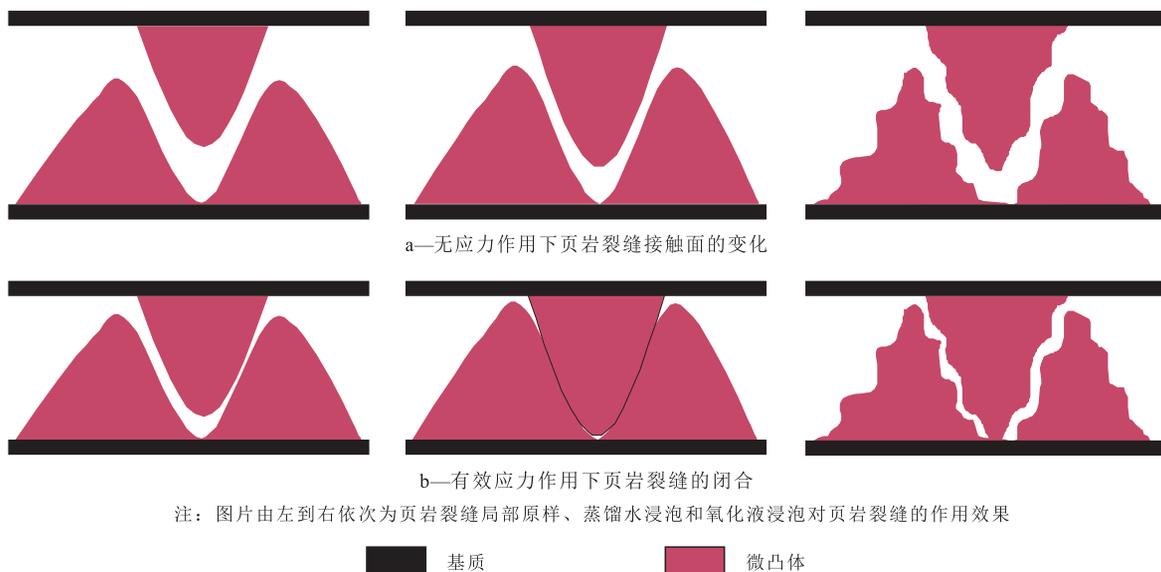
Fig.7 Scan electron microscope image of fractured samples after immersion in distilled water or in oxidization fluid

3.3 裂缝应力敏感性与页岩氧化增渗的关系

页岩层理和天然裂缝发育,水力压裂使天然裂缝网络开启,人工压裂裂缝和天然裂缝构成复杂的裂缝网络,是目前经济开发页岩气藏的必要条件。然而,页岩纳米孔供气速率低,压裂后气井难以达到高产目标,且短时间内产能下降快。利用页岩有机质、黄铁矿易氧化的特性,通过氧化溶蚀作用增

强孔隙连通性,且扩大孔隙尺寸,缩短基质气体传输时间,同时加速页岩微裂缝扩展延伸,提高压裂缝网密度,促进气体在基质与裂缝间的解吸和扩散,与水力压裂协同作用,实现对页岩基质的进一步改造,从而增强页岩微纳米孔缝的供气能力,减缓页岩气产能衰减速率。

一般页岩裂缝应力敏感性为中等偏强^[11],氧化液进入页岩裂缝网络后溶蚀裂缝面,影响页岩裂缝应力敏感性,直接关系到氧化增渗提升页岩气体传输能力的应用前景^[1-2]。分析此次研究结果认为,氧化溶蚀作用可扩大页岩基质纳米级孔缝,增强孔隙连通性^[4],有利于提高基质渗透率;另一方面,如图8所示,相比于页岩裂缝原样,蒸馏水浸泡后诱发粘土矿物层间水化膨胀,造成微凸体体积增大,在有效应力作用下加剧裂缝闭合^[12,27];而页岩中有机质、黄铁矿和碳酸盐矿物等发生氧化溶蚀,但石英、长石和粘土矿物含量稳定,导致裂缝面的不均匀溶蚀,减缓了裂缝的闭合,使得裂缝在有效应力作用下仍能保持相对较高的导流能力,裂缝的应力敏感性也随之降低。氧化溶蚀增强基质渗流能力,且未增强裂缝的应力敏感性,因而进一步论证了页岩氧化改造提升页岩气体传输能力的可行性。



注: 图片由左到右依次为页岩裂缝局部原样、蒸馏水浸泡和氧化液浸泡对页岩裂缝的作用效果

图8 氧化液浸泡后页岩裂缝接触面变化及裂缝闭合示意

Fig.8 Schematic diagram of surface change of shale fracture and its closure after immersion in oxidization fluid

4 结论

川东龙马溪组露头页岩岩样,经蒸馏水浸泡后渗透率提高0.42~6.15倍,应力敏感系数为0.514~0.587,应力敏感性为中等偏强;经氧化液浸泡后渗

透率提高5.53~65.45倍,应力敏感系数为0.482~0.517,应力敏感性有所减弱,为中等偏弱—中等偏强。通过对比氧化液浸泡前后岩样渗透率、矿物组分、截面宏观-微观形态特征的变化,以及分析浸泡后溶液的阳离子组成,发现裂缝面的选择性氧化溶蚀以及氧化溶蚀诱发的孔缝溶扩,是弱化页岩裂缝

应力敏感性的重要因素。针对常规水基压裂液导致页岩储层应力敏感性增强的问题,通过在压裂液中加入氧化液,利用氧化溶蚀作用提高基质渗透率且不改变甚至弱化裂缝应力敏感性的特性,以实现氧化改造提升页岩气体传输能力,进而达到提高页岩气藏采收率的目的。

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