

**THE ROLE OF ACIDIZING IN PROPPANT FRACTURING
IN CARBONATE RESERVOIRS**

A Thesis

by

JURAIKAT DENSIRIMONGKOL

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of
MASTER OF SCIENCE

August 2009

Major Subject: Petroleum Engineering

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ABSTRACT

The Role of Acidizing in Proppant Fracturing in

Carbonate Reservoirs. (August 2009)

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Chair of Advisory Committee: Dr. Ding Zhu

Today, optimizing well stimulation techniques to obtain maximum return of investment is still a challenge. Hydraulic fracturing is a typical application to improve ultimate recovery from oil and gas reservoirs. Proppant fracturing has become one of the most widely considered alternatives for application in carbonate reservoirs. Especially in areas that have high closure stress, the non-smoothly etched surface created by acid fracturing may not remain open upon closing, resulting in decrease in fracture conductivity and unsuccessful stimulation treatment.

In early years, because of the increase in the success of proppant fracturing, proppant partial monolayer has been put forward as a method that helps generate the maximum fracture conductivity from proppant fracturing treatment. However, this method was not widely successful because of proppant crushing and proppant embedment problems that result in losing conductivity. The ability to transport propping agents in available fracturing fluid was also poor and resulted in difficulties and failures to obtain proppant partial monolayer placement. For carbonate formations, acid

fracturing is another effective stimulation method. Simpler operation and lower cost made the technique attractive in the field with plenty of successful experiences. The heterogeneity feature of carbonate formation brings a challenge to create sufficient conductivity. In cases of high closure formation, fracture conductivity is hard to sustain. This factor limited the applications of acid fracturing sometimes.

In this study, laboratory tests were carried out using low concentrations of ultra-lightweight proppant to obtain partial monolayer proppant. Because of low specific gravity property of this proppant, it was claimed to help improve proppant transport inside the fracture.

In this experimental study, the partial monolayer technique was examined with particular emphasis upon the impact of acid in possibly improving fracture conductivity of carbonate rocks. The technique is referred as “closed fracture acidizing”. After obtaining a partial monolayer distribution on the fracture face, gelled acid was injected through the fracture face. Fracture conductivity before and after acid injection were evaluated.

Experimental results showed clearly that acid injection does not enhance fracture conductivity of partial monolayer proppant fracturing. The more the volume of acid injection, the more rapidly fracture conductivity declines.

DEDICATION

To my parents, and sisters for their unlimited support and encouragement
throughout this work

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CHAPTER I

INTRODUCTION

1.1 Literature Review

1.1.1 Hydraulic Fracturing in Carbonate Reservoirs

Hydraulic fracturing operation is applied to create highly conductive pathway in formations to enhance well productivity. The induced fracture tends to close because of the effect of the minimum horizontal stress. In acid-soluble formations, such as limestone, dolomite, and chalk, acid fracturing is usually performed to increase production rate and improve ultimate recovery.

In acid fracturing, hydrochloric acid is generally used to create the non-smooth etched surface which would leave open pathways to maintain fracture conductivity during the life of a well. The acid is injected at a pressure above formation fracturing pressure. In addition, wormholes and channel can be created to improve flow capacity in the formation. Fracture conductivity in acid fracture is generated by the pattern of the rock removal and the quality of rock removed. However, fracture conductivity does not necessarily increase as amounts of dissolved rock increases (Gong *et al* 1998; Abass *et al* 2006; Melendez *et al* 2007; Pournik *et al* 2007; Antelo *et al* 2008). Although the longer acid contact time with formation results in more rock dissolved, it lowers compressive strength of the formation. It may result in contact point failure, asperities embedment or asperities crushing which cause the fracture closure.

This thesis follows the style of *SPE Journal*.

Today proppant fracturing has become one of the most widely considered stimulation method for application in improving well performance, both for sandstone and also carbonate reservoirs. The main mechanism of proppant fracturing is to use non-reactive fracturing fluid to create a fracture deeply into the formation. Because the fracturing fluid is not reactive with the formation, the fracturing fluid can penetrate deeper compared to the acid fracturing. As a result, longer fracture can be anticipated from proppant fracturing. Proppant then is pumped down the fracture to hold the fracture open and to result in conductive pathway. The proppant fracturing is more favorable compared to acid fracturing to apply into some carbonate formations when

- Carbonate reservoir relatively homogeneous
- Acid solubility of the reservoir is low
- Field is located in high closure stress area
- Rock softens significantly under closure after contact with acid

Relatively homogeneous carbonate reservoir

For homogeneous carbonate reservoir, acid fracturing possibly creates a more uniform pattern of etched surfaces on the fracture faces. The uniform etched surface does not have sufficient roughness to provide open channels after fracture close because of closure stress resulting in unsuccessful acid fracturing treatment.

Low acid solubility of the reservoir

There would be insufficient amount of rock dissolved in which the wormholes and channels cannot be created. As a result, the fracture conductivity from acid fracturing treatment would not improve well production efficiently.

High closure stress formation

The non-smoothly etched surface created by acid fracturing cannot support such a high closure stress and the fracture may not remain open upon closing, resulting in decrease in fracture conductivity and unsuccessful acid stimulation treatment.

Soft formation

For soft rocks, after contact with acid, fracture strength is not sufficient to withstand minimum horizontal stresses because of compressive failure of contact points on the etched surface.

1.1.2 Proppant Partial Monolayer Technique

Proppant partial monolayer was claimed to be a recovery method that maximizes conductivity in proppant fracturing. Darin and Huitt (1959) explored the potential advantage of partial monolayer over a pack of propping agents. A laboratory study was set up to determine the flow capacity of a fracture with a partial monolayer distribution. Light hydrocarbon oil was used as a flowing medium through propping agent ball. From their experimental results, the flow capacity of partial monolayer proved to be an order of magnitude greater than the flow capacity provided by a full monolayer and multipack of proppant.

As shown in **Fig. 1.1**, proppant particles are closely packed in full monolayer proppant, but there are vacant areas around and between proppant particles in a fracture containing partial monolayer proppant. Because of these vacant areas, partial monolayer provides more flow capacity and resulting in higher fracture conductivity.

Proppant transport is one of the key parameters in achieving partial monolayer distribution of propping agents. Harrington and Hannah (1975) cited the reason why the partial monolayer technique was abandoned. The ability to transport propping agents in available fracturing fluid was poor, resulting in the difficulties and failure to obtain proppant partial monolayer. In addition, insufficient proppant strength resulting in proppant crushing, and losses of fracture width because of proppant embedment were critical concerns causing this technique unsuccessful.

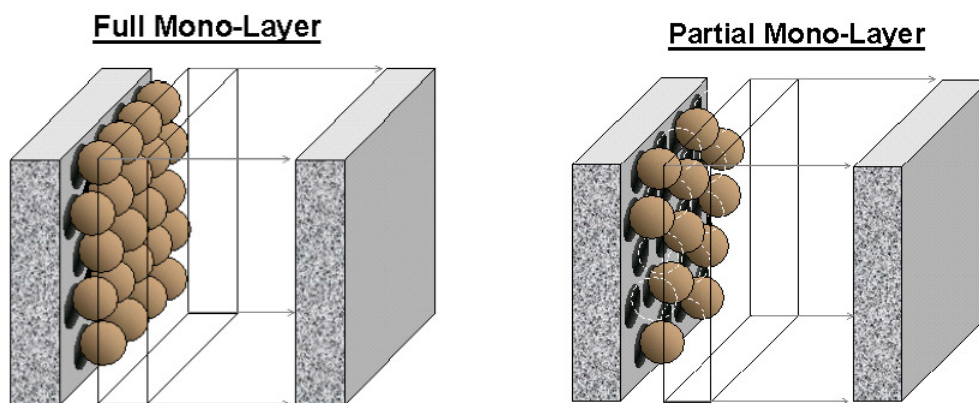


Fig. 1.1—Representation of a fracture containing a full monolayer and a partial monolayer (Brannon *et al* 2004)

Gidley *et al* (1989) emphasized that the early industry interested in placing partial monolayer to obtain maximum conductivity, but there has no prove to be a successful concept. Main reason was the lack of ability to achieve uniform and complete coverage of the fracture with partial monolayer.

Brannon *et al* (2004) described parameters controlling proppant settling velocity based upon Stoke's Law. The proppant settling velocity (V) in ft/min can be described as

$$V = 1.15 \times 10^3 \left(\frac{d_p^2}{\mu_{fluid}} \right) (\gamma_{prop} - \gamma_{fluid}) \quad (1.1)$$

where,

d_p	=	Median Proppant Particle Diameter (in)
V	=	Proppant Settling Velocity (ft/min)
μ_{fluid}	=	Fluid Viscosity (cp)
γ_{prop}	=	Proppant Specific Gravity
γ_{fluid}	=	Fluid Specific Gravity

As slickwater fracturing has proven to be a cost-effective well stimulation technique, the size and specific gravity of the propping agent has become even more critical in relation to proppant settling velocity. Lighter propping agents tend to fall slower than heavier ones. They performed slot flow testing and the results showed significant reduction in settling velocity of ultra-lightweight proppant which has 1.25 specific gravity compared to the 20/40 Ottawa sand. As a result, an ultra-lightweight proppant had been claimed to improve proppant transport and assist in achieving monolayer distribution of propping agents in a fracture. They also conducted a series of experiments on sandstone to investigate fracture conductivity characteristic of partial

monolayer at different proppant concentrations using ultra-lightweight proppant. The conductivities of partial monolayer were found to be an order of magnitude greater than similarly sized sands. However, proppant was loaded manually instead of dynamically pumping through the fracture.

Chambers and Meise (2005) argued that a partial monolayer can be generated through the use of low concentrations of ultra-lightweight proppant. They contended that the technique resulted in a significant production gain.

1.1.3 Closed Fracture Acidizing Technique (CFA)

Closed fracture acidizing technique (CFA) has been introduced as a viable technique to increase final conductivity. A small acid stage is pumped through a closed or partially closed fracture at below fracturing pressure. When acid flows through this closed fracture, possibly in turbulent flow, rapidly dissolving more rock on the fracture face can be anticipated than flowing in an open fracture. Because of the heterogeneity in most carbonate formation, one area in which has more acid solubility or higher permeability might dissolve faster than an adjacent area. That dissolved area becomes larger in a very short period. Most of acid tends to flow through this area, so channels or grooves can be created. Not only natural fracture formation, but also created fracture can be pumped through using acid to dissolve more soluble material on the fracture face and increase flow capacity. This technique has been only studied for the created fracture from acid fracturing treatment.

Fredrickson (1986) examined the effect of acid on flow capacity of an acid etched fracture using 2.25 to 4 in. circular disk. The experimental results showed that the closed fracture acidizing technique will not improve flow capacity on certain types of formations. He suggested that this technique should be applied in any carbonate formation, stating that fracture face should not soften too much so that it can sustain fracture conductivity after closing.

Bartko *et al* (1992) presented experimental data suggesting substantial gains in conductivity of increasing orders of magnitude after implementing the closed fracture acidizing treatment to the created fracture resulting from an acid fracturing operation.

Kalfayan (2007) suggested that the closed fracturing acidizing technique may not develop sufficient etched fracture conductivity if the formation is too soft, as in chalks. The etched flow channels may be crushed or embedded upon the closure.

1.2 Problem Description

The fracture conductivity is a key factor to determine the success of a stimulation treatment. Lower conductivity can lead to lower well productivity and potentially lead to economic failure. Flow capacity inside the fracture after fracture closure is a significant parameter to control final fracture conductivity. To increase the flow capacity of a fracture, many techniques have been investigated and studied.

Acid is sometimes injected into carbonate formations to improve flow capacity of a created fracture. Although fracture conductivity of a created fracture is anticipated to improve after acidizing, acid can sometimes soften the rock too much that the

fracture cannot sustain high closure stress upon closing, resulting in decrease in final conductivity. Therefore, it is required a thorough understanding of the influence of the acid treatment variables on each particular type of formation.

In this study, a series of experiments is performed to study the effect of the acid on fracture conductivity in a closed fracture from proppant fracturing. **Fig. 1.2A** shows the flow area improvement of proppant partial monolayer fracturing after acidizing. The red shaded area in **Fig 1.2B** represents the flow area gained after acid injection. In the past, there was no study focusing on the effect of the acid injection in a propped fracture. Only the created fracture from acid fracturing was examined.

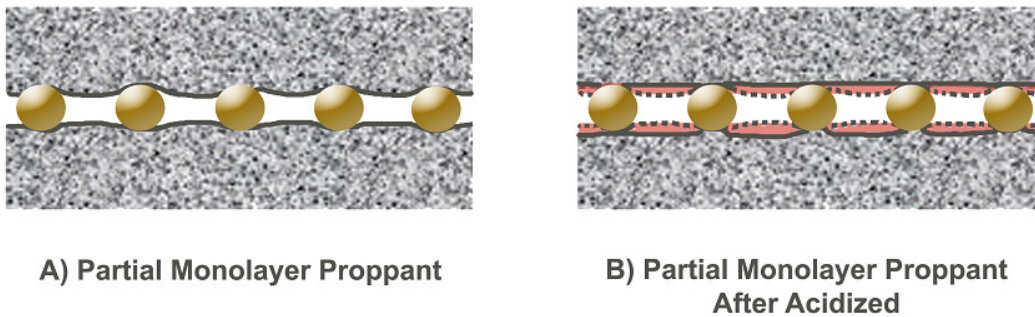


Fig. 1.2-Representation of a fracture containing a partial monolayer proppant and a partial monolayer proppant after acidizing

1.3 Research Objective

This research examines and identifies the effect of the acid on fracture conductivity in a closed fracture with partial monolayer propping agents, based on experimental work. A profilometer device is used to characterize the surface profile after acidizing. The effect of acid contact time and acid injection rate on the final conductivity is evaluated.

This study provides a better understanding of the closed fracture acidizing technique on fracture conductivity of partial monolayer proppant fracturing, which aids in the future design of fracturing design treatment for carbonate reservoirs and serve as valuable information to consider for future work.

CHAPTER II

EXPERIMENTAL SET UP, PROCEDURES, AND CONDITIONS

2.1 Experimental Apparatus

The laboratories for dynamic proppant pumping and acid fracturing were used in this study. The dynamic proppant pumping was designed with a goal to load the proppant through pumping, which is more representing the actual field conditions. In addition, it was designed to develop the dynamic fracture conductivity testing to provide appropriate scaling field conditions. For acid fracturing apparatus, it was designed to deal with the required conditions for experimentation on different flow rates similar to field condition in acid fracturing treatment.

2.1.1 Dynamic Fracture Conductivity

Equipment and piping in this laboratory were designed to handle only non-corrosive fluid and material. The dynamic fracture conductivity procedure can be divided into two parts:

- Dynamic proppant pumping
- Fracture conductivity measurement

Dynamic Proppant Pumping

The dynamic proppant pumping apparatus was designed to simulate a propped fracture by pumping a fracturing fluid with proppant through 2 pieces of core samples.

The schematic of the apparatus for fracturing fluid pumping is shown in **Fig. 2.1**. A mixing tank is used to prepare fracturing fluid which is a mixture of tap water and proppant. The slurry was pumped at 4 gal/min in all experiments through the horizontal fracture. Hydraulic load frame is used to provide the closure stress on core samples. The loading frame can apply up to 25,000 psi closure stress. It has a ram area of 125 in², so there is about 10 times the force applied to the load frame is actually acting on the core samples which have a ram area of 12.2 in². All experiments were performed at room temperature.

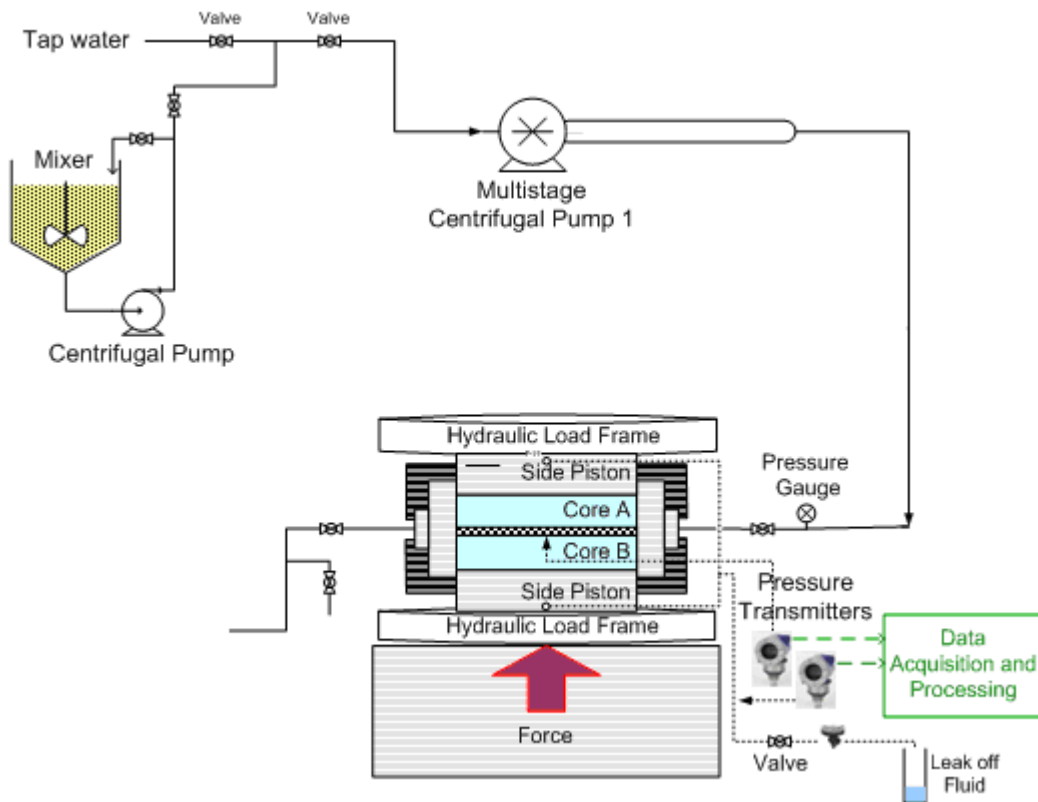


Fig. 2.1—Schematic of dynamic proppant placement

Fracture conductivity measurement

The schematic of the fracture conductivity measurement is shown in **Fig. 2.2**. Nitrogen gas was used for conductivity measurement. The detailed discussion of dynamic fracture conductivity test is discussed by Marpaung (2007). To simulate the wet gas condition, a water chamber was used. Conductivity was measured by flowing wet N_2 gas into the proppant partial monolayer packed inside the fracture. Two pressure transducers were used to measure cell pressure and pressure drop across the fracture face. Closure stresses from 500 psi in increases of 500 psi are applied to measure the pressure drop across the fracture until the fracture is closed. At each closure stress, five different gas flow rate were used to measure the pressure drop. A nitrogen flow regulator was used to control the gas flow rate during conductivity measurement.

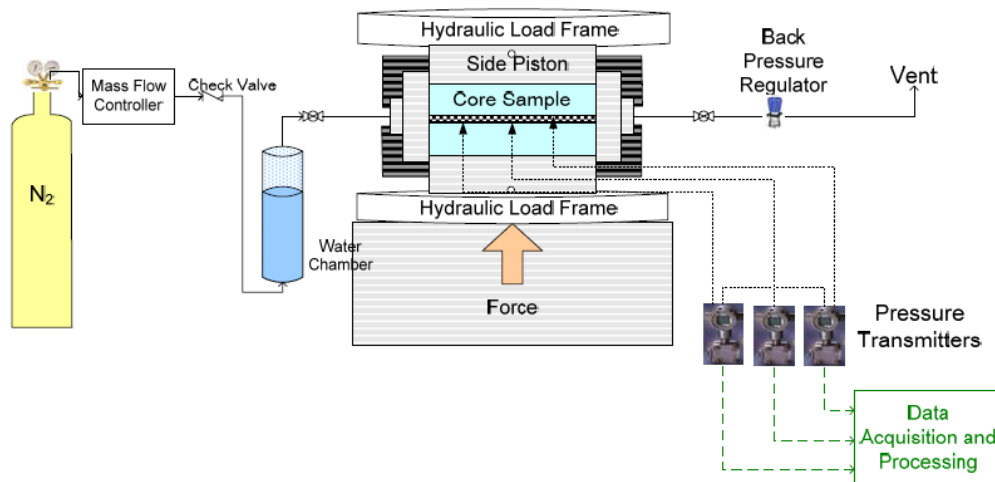


Fig. 2.2—Schematic of fracture conductivity measurement (After Marpaung 2007)

2.1.2 Acid Fracturing

Apparatus and flow line in this laboratory were designed to handle corrosive fluid. The acid fracturing procedure can be divided into two parts:

- Acid injection
- Surface characterization

Acid injection

Fig. 2.3 shows the apparatus for acid injection process to simulate closed fracture acidizing application. There are three accumulators using for our experiments. Two of them are made of a corrosion resistant material which is Hastelloy material. Both of them are designed to use for acid with capacity of 1000 ml. Another one accumulator is made of stainless steel which has a capacity of 4000 ml. This stainless steel accumulator was used to store water. The displacement of the acid and water is performed by the syringe pumps. The syringe pump is used to pump the hydraulic oil to press onto the Teflon piston inside the accumulators pushing either acid or water out at a certain pressure and flow rate being set on the pump. In our experiment, the inject rate were 20 and 30 ml/min. PVC refill container is used to refill the piston accumulator. Either acid or water is filled in the PVC container first. Then air pressure provided from the laboratory air system is applied in the container at 100 psi to push the fluid into an accumulator.

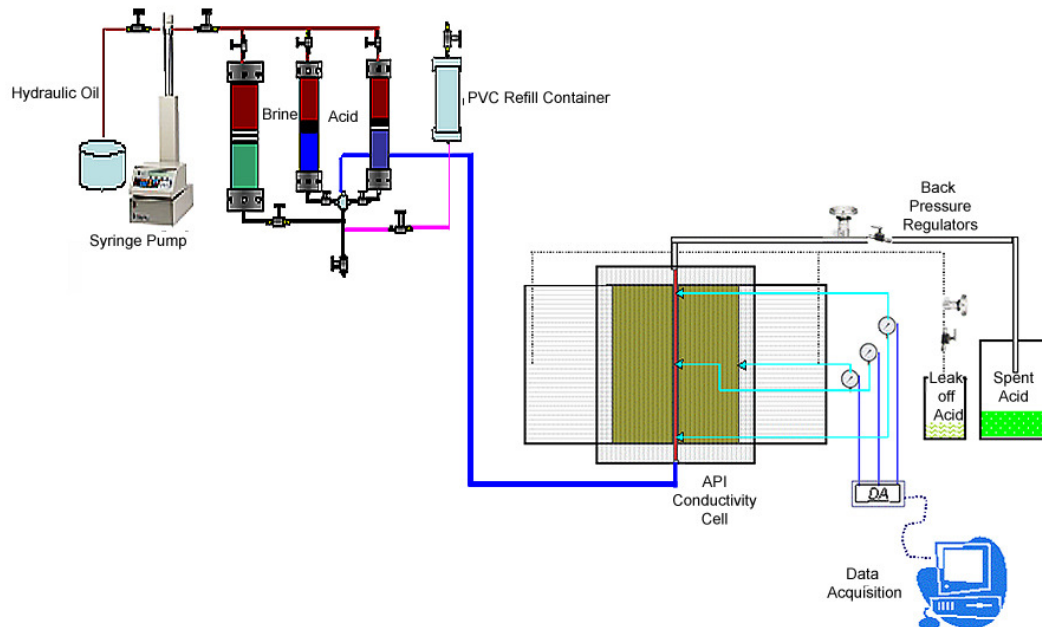


Fig. 2.3—Schematic of closed fracture acidizing

The conductivity cell was placed vertically during acidizing to avoid gravity effect. The cell pressure was kept constant at above 1000 psi by using back pressure regulator to ensure that CO_2 generated from the acid reaction is miscible in the solution. Moreover, the 500-psi closure stress was applied on created fracture during acid injection to avoid freely move of proppant. The pressure cell is connected to three pressure transducers to monitor the experimental conditions during the test. There are three pressure transducers using in this experiment. One pressure transducer monitors cell pressure, another one measures the pressure drop across the fracture face and the

other observes the leakoff pressure. In our experiment, we kept zero leakoff pressure. The details of acid fracturing test equipment are presented by Melendez (2007).

Surface characterization

The profilometer apparatus (**Fig 2.4**) is used to characterize the surface profile of the rock. Detailed description of the profilometer is presented by Nieto (2007). A profilometer is a precision vertical distance measurement device which can measure small surface variations in vertical surface topography as a function of the surface position. The vertical measurement is made with a laser displacement sensor while the sample is moved along its length on a moving table. That measurement is repeated several times over the width of the sample to cover the entire surface area.

In our experiment, the surface scanning was performed after acid injection. The surface profile of the etched surface with proppant partial monolayer was investigated in a relation of fracture conductivity after the acidizing.

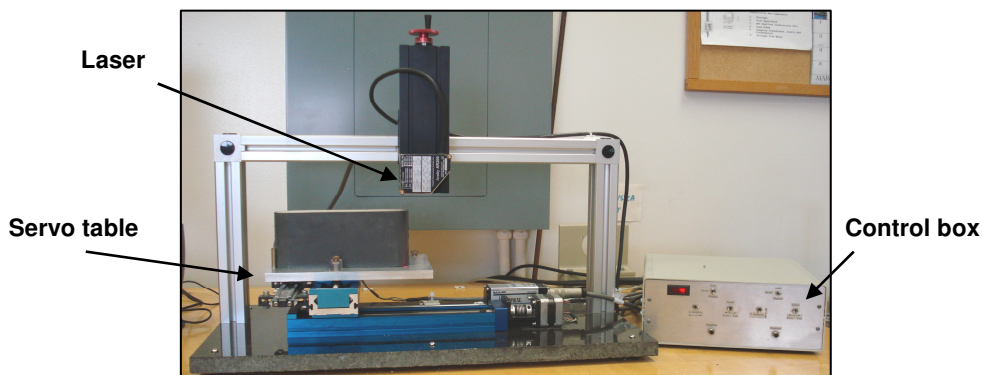


Fig. 2.4—Profilometer device

2.1.3 Test Cell

The test cell for dynamic proppant pumping and acid fracturing is made of Hastelloy material which is acid resistant. This test cell is a modified API RP-61 conductivity cell. Dimensions of the cell body are 10 in. long, 3-1/4 in. wide and 8 in. height. **Fig. 2.5** shows the conductivity cell and a core sample used for experiments. The conductivity cell had a special internal structure consisting of a rounded edge to accommodate the rock samples. Internal part of the pressure cell was also equipped with two o-rings to avoid leaking between a rock sample and the wall of the test cell. The rock samples used in this study had a rectangular shape with rounded edges to provide the best fit of the core inside the cell.

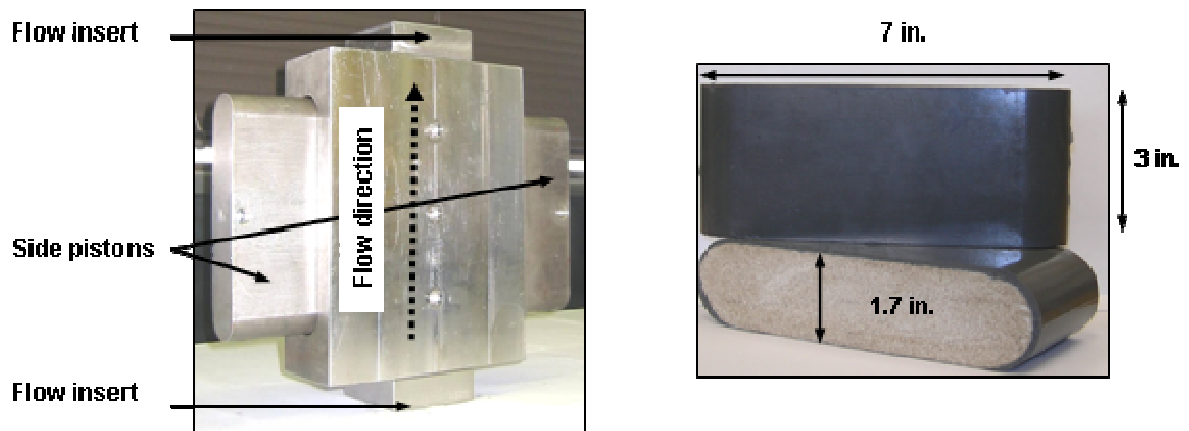


Fig. 2.5—Conductivity cell and core sample used for experiments

Dimensions of core samples are 7 in. long, 1.7 in. wide and 3 in. height. The core samples were covered with a sealant material to provide a perfect fit inside the cell.

Side pistons with o-rings on the edges are used to keep the cores in place during the experiment. Flow inserts in the bottom and upper surface of the cell are attached to connect the flow lines. There are three access ports at one side of the cell body to connect to the pressure transducer for pressure measurement.

2.2 Experimental Procedure

The experimental procedure consists of seven steps as shown in **Fig. 2.6**. The description of each step is listed below.

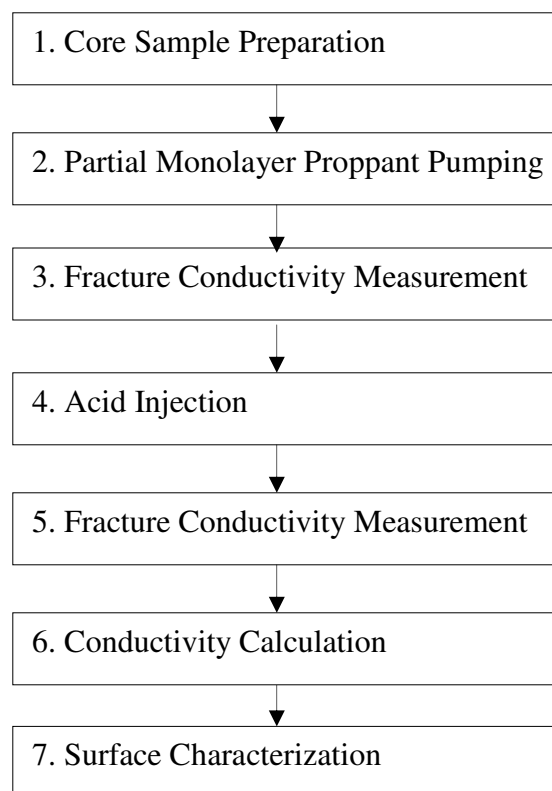


Fig. 2.6—Experimental process for proppant fracturing and acid fracturing

2.2.1 Core Sample Preparation

Indiana limestone was used in this experiment to study the effect of the acid on fracture conductivity of partial monolayer proppant. The rock samples were cut to a rectangular shape with round edges with a length of 7 in., a width of 1.7 in. and a height of 3 in. using an electric cutter machine. Core samples were covered with a silicone-base sealant to provide a perfect fit of the core samples inside the conductivity cell. Core samples before and after covered with silicone rubber are shown in **Fig. 2.7**.

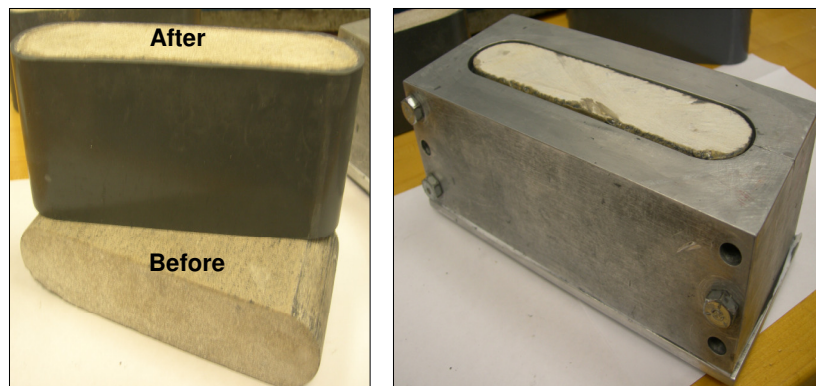


Fig. 2.7—Core samples and mold used to prepare the core samples

The procedures to prepare the core samples are as follows.

1. Put blue tape on the top and bottom of a core sample, cutting edges with razor cutter.
2. Apply the edges of the rock surfaces with the silicone primer (SS415501P) three times. Allow 15 minutes waiting time in between primer applications.

3. Clean metal surface and bottom plastic part of mold with cloth and stoner spray.
The mold structure used is showed in **Fig. 2.7**. The mold is made of stainless steel, with a plastic bottom.
4. Assemble the mold. Tighten the four bottom screws and the three side screws.
Make sure all bolts are tight.
5. Put the rock in the mold and adjust the center position.
6. Mix silicone potting compound and silicon curing agent for 1:1 mixing ratio.
Weigh before mixing both components to ensure that the mixture is 50/50 of each component, either by volume or by weight percent. Mix and stir it thoroughly.
7. Use a disposal injection system to pour mixture in the mold carefully until the silicone fills to the top of the rock sample.
8. Remove the top duct tapes and put the molds into the oven at 100°C for approximately 1 hour.
9. Remove the molds from the oven and wait for two (2) hours until the molds temperature decreases.
10. Unscrew all the bolts from the mold and carefully remove the samples from the mold.
11. Cut extra silicon on the edges with a razor cutter.
12. Label the rock sample. The core sample is ready to use.

2.2.2 Partial Monolayer Proppant Pumping

Dynamic proppant pumping apparatus was used to place the partial monolayer distribution on the fracture face before acidizing. The detail procedure for proppant fracturing to obtain partial monolayer proppant is as follow.

1. Prepare the core samples. Follow the guideline in section 2.2.1.
2. Put two O-rings in the grooves inside the conductivity cell.
3. Insert the bottom core sample into the conductivity cell using hydraulic jack.
4. Insert the top core sample into the conductivity cell. Put a shim which the shim thickness is equal to the desired fracture width in between the top and the bottom core samples. In our case, we used 0.374 in. fracture width before start pumping the fracturing fluid.
5. Put the conductivity cell into the support rack. The support rack is used to keep one closure stress on the fracture face during the experiment.
6. Put the conductivity cell with the support rack in the center of the hydraulic load frame.
7. Activate the AP-1000 hydraulic pump by opening the air supply valve. Open the air regulator and adjust the supply pressure to move the bottom ram of the hydraulic load frame until the top of the support rack touches the top plate. Then close the air regulator.
8. Connect all pumping lines into the conductivity cell.

9. Put the desired amount of water and proppant into the mixing tank. In our experiment, ultra-lightweight proppant was used to achieve 0.02 lb/ft^2 on the fracture face.
10. Pump the slurry through the fracture face for 10 seconds then immediately close the inlet and the outlet valves and open the bypass valve.
11. Open the valve located on the top piston while applying closure stress gradually to close the propped fracture. It allows excess water to drain out when closing the fracture. Close the air regulator for the load frame when the closure stress reaches 500 psi. Then disconnect pumping inlet and outlet lines.

2.2.3 Closed Fracture Acidizing

Acidizing apparatus was used to inject acid into a closed fracture with proppant partial monolayer. In our study, low reaction rate gelled acid was used. The detail procedure for acid injection is listed below.

1. After obtaining partial monolayer distribution of the propping agents on the fracture face, the conductivity cell must be kept under 500 psi closure stress at all time by using the support rack during acidizing to prevent a propping agent to be freely move inside the fracture and to simulate the field condition.
2. Put the cell with the support rack onto the acidizing stand.
3. Connect the inlet and outlet lines onto the conductivity cell. Then connect all pressure lines to the access ports to measure pressure during acidizing.

4. Prepared gelled acid and fill into one Hastelloy accumulator using PVC refill container. The acid composition and concentrations is shown in section 2.3.2.
5. Refill 4000 ml stainless steel accumulator with water using PVC refill container and air pressure. It is recommended to fill water accumulator until it is full since water need to use for both pre and post acid injection. Then refill syringe pump with hydraulic oil.
6. Set back pressure regulator at the outlet line to 1000 psi.
7. Inject water into the conductivity cell at a desired flow rate until the cell pressure reaches 1000 psi.
8. After obtaining 1000 psi cell pressure, switch to inject gelled acid until the desired contact time. Maintain cell pressure at 1000 psi or above at all time to ensure that CO₂ is miscible in the solution to simulate field conditions. Flow rates and contact times will be varied from one set of experiments to another.
9. Change the flow from acid to water from water accumulator when the desired testing time is completed. Flush lines with water until pH values from the outlet are values between 7 and 7.5.
10. Lower cell pressure gradually to zero, then turn off the pump.
11. Disconnect the inlet, outlet and pressure lines from the conductivity cell.
12. Take out the cell with the support rack from acidizing stand. Then take it to the conductivity measurement laboratory.

2.2.4 Fracture Conductivity Measurement

Low pressure Nitrogen gas is used to measure fracture conductivity and simulate gas reservoir condition. The conductivity before and after acid treatment were measured and evaluated to determine the effect of the acid. The procedures to measure conductivity are as follows:

1. Put the conductivity cell with the support rack in the center of the hydraulic load frame.
2. Release all locks on the support rack to release the closure stress.
3. Adjust the pressure on AP-1000 hydraulic pump to obtain 500 psi closure stress acting on the fracture.
4. Calibrate the mass flow controller to zero point by adjusting flow controller to the closed position and wait until the reading is zero.
5. Connect the inlet to the nitrogen line and the outlet to the back pressure regulator.
6. Open the nitrogen regulator and mass flow controller to flow gas into the conductivity cell.
7. Check all lines for leakage. Close the nitrogen regulator if leakage is found and repair the leak.
8. Adjust nitrogen regulator, back pressure regulator, and mass flow controller until the cell pressure reaches a value of 50 psi.
9. Wait until flow rates and pressure readings stabilize and record the gas flow rate, cell pressure, and differential pressure. Record the cell pressure and pressure drop across the fracture.

10. Increase the flowrate and record its corresponding pressure drop. Repeat the readings at 5 different flowrates. When increasing the flowrate, cell pressure will increase. Adjust the backpressure regulator to decrease cell pressure to 50 psi for each reading.
11. Increase closure stress by 500 psi. Repeat and record the pressure drops for different flowrates until the back pressure can no longer control the cell pressure or the pressure drop in the fracture is higher than the allowable range of the pressure transducer.
12. Turn off the nitrogen flow and disconnect all lines to the conductivity cell.
13. Lower the load frame pressure to allow the removal of the conductivity cell. Then take off the support rack.
14. Remove the rock sample from the cell with the hydraulic jack.

2.2.5 Fracture Conductivity Calculation

To calculate the fracture conductivity data from the experimental data, the Forcheimer equation is arranged to obtain a straight line equation in which $\frac{\rho q}{\mu h}$ is the x-axis and $\frac{(p_1^2 - p_2^2)Mh}{2ZRTL\mu\rho q}$ is the y-axis. The y-intercept is the inverse of the fracture conductivity.

$$\frac{(p_1^2 - p_2^2)Mh}{2ZRTL\mu\rho q} = \frac{1}{k_f w} + \frac{\beta\rho q}{w^2\mu h} \quad (2.1)$$

The pressure drop (p_1 and p_2) were measured in the lab at five different flowrates under each closure stress. **Table 2.1** shows the values of all the other variables we used in the fracture conductivity calculation.

TABLE 2.1—DATA USED FOR CONDUCTIVITY CALCULATION

M	Molecular mass of nitrogen, kg/kg mol	0.028
h	Height of fracture face, in	1.61
Z	Compressibility factor	1.00
R	Universal constant, J/mol K	8.3144
L	Length of fracture over which pressure drop is measured, in	5.25
μ	Viscosity of nitrogen at standard conditions, Pa.s	1.759E-05
ρ	Density of nitrogen at standard conditions, kg/m ³	1.16085

2.2.6 Matrix Flow Calculation

To calculate the matrix flow, we applied Forcheimer's equation (Eq. 2.1) and Darcy's law.

$$q_{sc} = \frac{kA}{2\mu p_{sc}} \frac{(p_1^2 - p_2^2)}{L} \quad (2.2)$$

By neglecting the non-Darcy flow term, Eq. 2.1 and Eq. 2.2 can be rearranged to calculate the matrix flow conductivity.

$$k_f w = \frac{ZRT\rho}{p_{sc}} \frac{kA}{Mh} \quad (2.3)$$

Table 2.2 shows the additional variables we used in the matrix flow calculation.

After applied Eq. 2.3 to calculate the matrix flow conductivity, the value of the matrix flow conductivity for Indiana limestone is approximately 2 md-ft.

TABLE 2.2—ADDITIONAL DATA USED FOR MATRIX FLOW CALCULATION

A	Cross section area in the flow direction, cm ²	63.871
k	Permeability of Indiana limestone, md	4
p _{sc}	Absolute base pressure, atm	1

2.3 Experimental Conditions

In this experimental study, Indiana limestone was used to investigate the effect of the acid on the fracture conductivity of partial monolayer proppant fracturing. The proppant fracturing was simulated in the dynamic proppant pumping laboratory to achieve partial monolayer distribution of the propping agent in the fracture face. Tap water was used as a fracturing fluid. For all experiments, the fracturing fluid with proppant was pumped at 4 gallons/min at room temperature. The following is the detail of proppant and acid used in this experiment.

2.3.1 Proppant Size and Concentration

The ultra-lightweight proppant with 14-40 mesh size was used in this experimental study. This proppant has specific gravity of 1.05 which approaches neutrally buoyancy with water. Low proppant concentration (0.02 lb/ft²) was used in all experiments to achieve proppant partial monolayer inside the fracture. Since we will

not study the effect of proppant size and proppant concentration, only one proppant size and concentration were used to achieve the objective of this research.

2.3.2 Acid Composition and Concentration

A gelled acid with 15% HCl was selected for this experiment. The gelled acid was injected at room temperature for all experiments. The composition of gelled acid used for this study is shown in **Table 2.3**.

TABLE 2.3- GELLED ACID COMPOSITIONS AND CONCENTRATIONS

Chemical	Concentration
Concentrated HCl acid (31%), ml/liter	448
Water, ml/liter	522
Corrosion inhibitor, ml/liter	3
Iron control, ml/liter	10
Gelling agent, ml/liter	15
Surfactant, ml/liter	2

The additives components for the selected acid are as follows.

1. Corrosion inhibitor is used to prevent corrosion of the metal. It works by absorbing negative charges on metal surface and build a layer on metal to isolate acid from metal.
2. Iron control is used to decrease amount of iron in the solution to prevent the iron precipitation during and after acid reaction.
3. Gelling agent is a type of polymer which is used to increase the viscosity of acid and also to decrease the acid reaction rate.

4. Surfactant removes oil or hydrocarbon coated on rock surface which allow acid to contact and react with rock mineral.

2.4 Experimental Output

2.4.1 Conductivity Values

Forcheimer equation is used to estimate fracture conductivity for both before and after acidizing. The cell pressure and pressure drop were recorded at each flow rate under each closure stress. By calculating fracture conductivity as the section 2.2.6, the conductivity profile can be evaluated the effect of the acid on the fracture conductivity.

Fig. 2.8 presents an example of conductivity profile before and after acidizing for 20 ml/min acid injection rate and 10 minutes contact time. The conductivity value before acid treatment represents fracture conductivity of a partial monolayer proppant fracturing application. The conductivity measurement was performed until the closure stress reached 1500 psi to avoid the damage of the proppant and fracture face by higher closure stress before acidizing. The fracture conductivity after acidizing was then measured and compared with the fracture conductivity before acid injection.

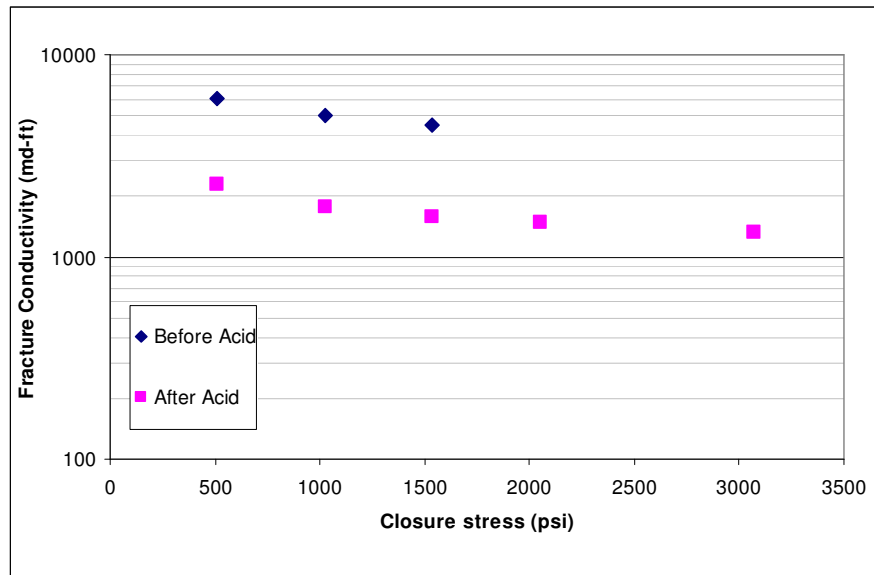


Fig. 2.8—Conductivity before and after acidizing for 20 ml/min acid injection rate and 10 minutes contact time

2.4.2 Surface Profile

Profilometer generated 3D images which represents the surface profile of the rock after acidizing. The images are represented with a color scale, which corresponds to depth of dissolution, with values ranging from -0.008 to 0.008 inches. The values increase from a darker shade of blue to a red shade. Examples of the images generated are shown in **Fig. 2.9**.

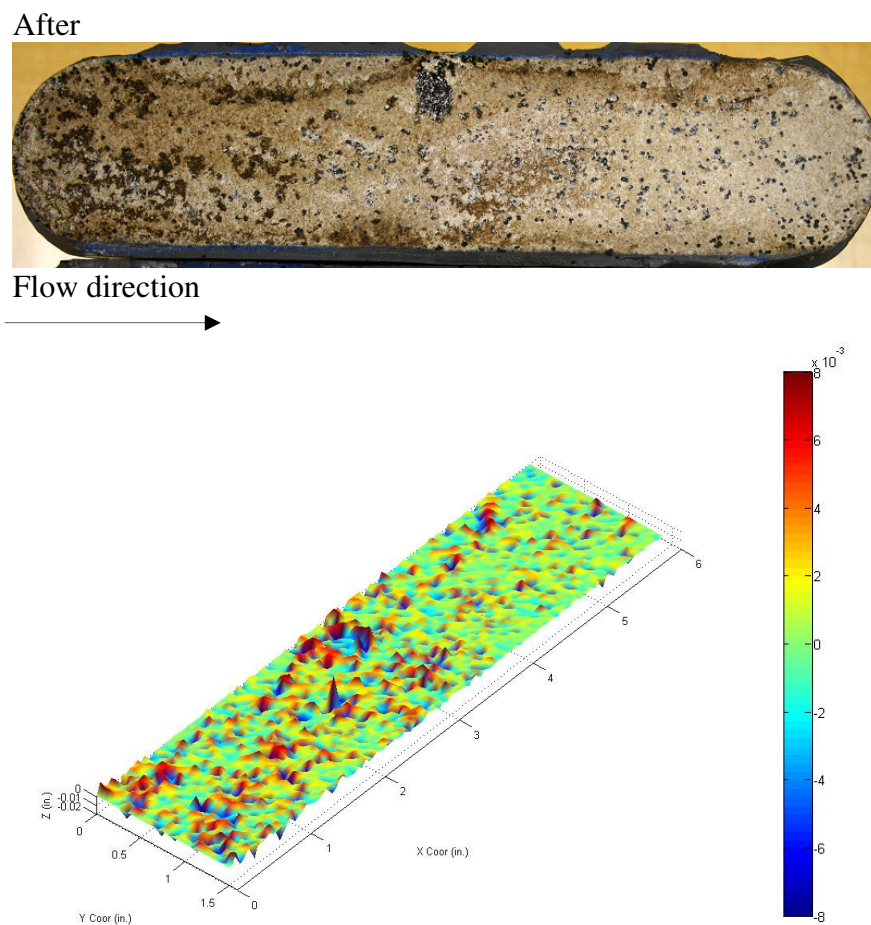


Fig. 2.9—Photograph and 3D surface image of a core sample after acidizing (20 ml/min and 15 mins contact time condition)

CHAPTER III

EXPERIMENTAL RESULTS AND DISCUSSION

A series of experiments was conducted using Indiana limestone by obtaining partial monolayer using ultra-lightweight proppant before injecting a gelled acid system with different acid injection rates and contact times at room temperature. The experiment of 20 ml/min injection rate and 10 minutes contact time was repeated to evaluate the consistency of the experimental procedure. All experimental conditions of this study are summarized in **Table 3.1**. The experimental data including photographs, and 3D surface profiles are presented in Appendix.

TABLE 3.1—SUMMARY OF EXPERIMENTAL CONDITIONS

Test No.	Acid Injection Rate (ml/min)	Acid Contact Time (mins)	Acid Injection Volume (ml)
1	Proppant Fracturing only, No acid		-
2	20	10	200
3	20	10	200
4	20	15	300
5	20	20	400
6	30	10	300

3.1 Fracture Conductivity Results

Fracture conductivity results of all experimental conditions are listed in **Table**

3.2.

TABLE 3.2—SUMMARY OF CONDUCTIVITY VALUES OF ALL EXPERIMENTS

Test No.	Acid Injection Rate (ml/min)	Acid Contact Time (mins)	Closure Stress (psi)					
			512	1,025	1,537	2,049	2,561	3,074
1	Proppant Fracturing only, No acid		4960	3267	2338	1577	1251	534
2	20	10	3538	3006	2208	1585	-	-
3	20	10	2292	1766	1588	1482	1335	-
4	20	15	1752	1214	771	479	-	-
5	20	20	1517	963	440	207	112	-
6	30	10	3936	2662	1165	690	-	-

The experimental data showed that the fracture conductivity of partial monolayer proppant fracturing without acid treatment is higher than all the other tests. From the results, it suggests that acid treatment after obtained partial monolayer distribution in the fracture did not help improve fracture conductivity, but decrease in conductivity.

3.1.1 Consistency of Experimental Procedure

The experimental repeatability can assist in determining the consistency of the experimental procedure. The 20 ml/min injection rate with 10-min contact time experimental condition was repeated as shown in Test 2 and Test 3 experimental results.

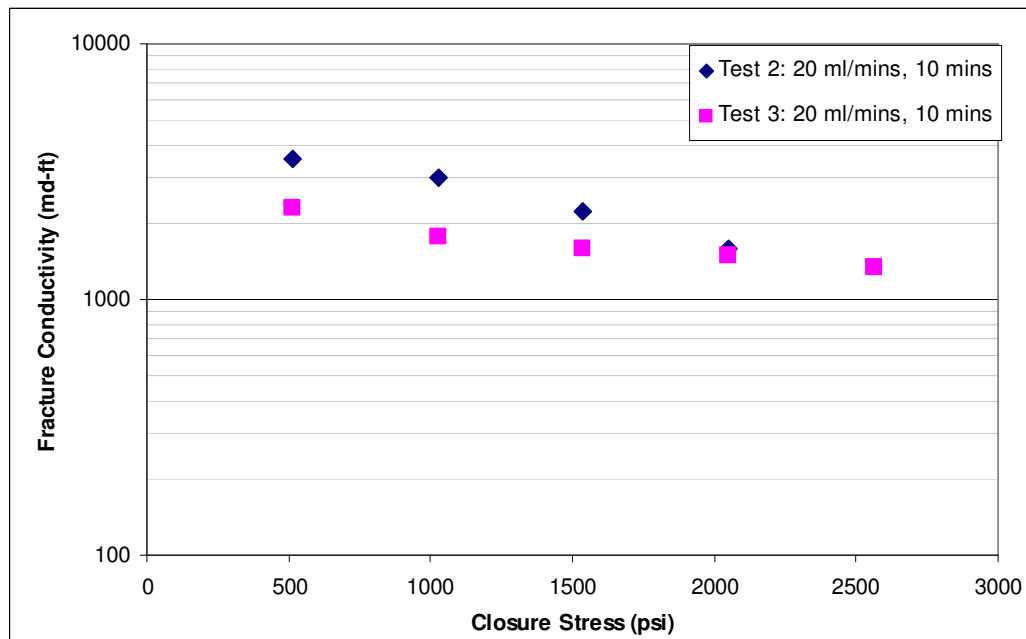


Fig. 3.1—Experimental repeatability result to determine consistency of the experimental procedure

Fig. 3.1 shows the conductivity results from two tests which have the same experimental condition (20 ml/min and 10-mins contact time). Both Test 2 and Test 3 provided almost similar fracture conductivity values with stable fracture conductivity reduction trend. These results show that the experimental technique using in this study is reliable and provides us the consistent results.

3.1.2 Effect of Acid Contact Time

Acid contact time is a critical factor that affects the final conductivity. Comparison of the experimental results at same injection rate but different contact time can help evaluate the effect.

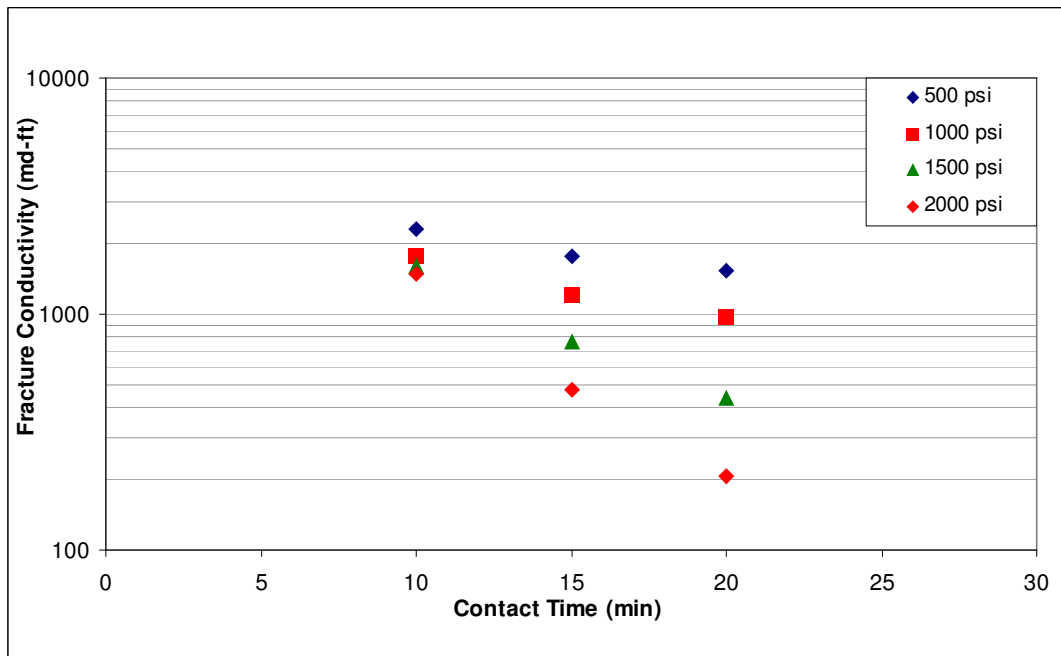


Fig. 3.2—Effect of contact time on fracture conductivity for 20 ml/min injection rate at four different closure stresses

The results for 20 ml/min injection rate are plotted in **Fig 3.2** indicating the effect of acid contact time. The trend of decreasing the fracture conductivity with increasing acid contact time shows in all closure stress conditions. At 500-psi closure stress, the conductivity decreases gradually with increased acid contact time, ranging from approximately 2,300 md-ft for the 10-min. contact time experiment to approximately 1,500 md-ft for the 20-min test. In addition, with 2,000-psi closure stress, the conductivity declines more rapidly from approximately 1,500 md-ft for 10-min test to 200 md-ft at 20-min test. The results suggest that longer acid exposure time yield lower fracture conductivity than can be obtained with shorter acid contact times and this effect becomes more important at the higher closure stress.

3.1.3 Effect of Acid Injection Rate

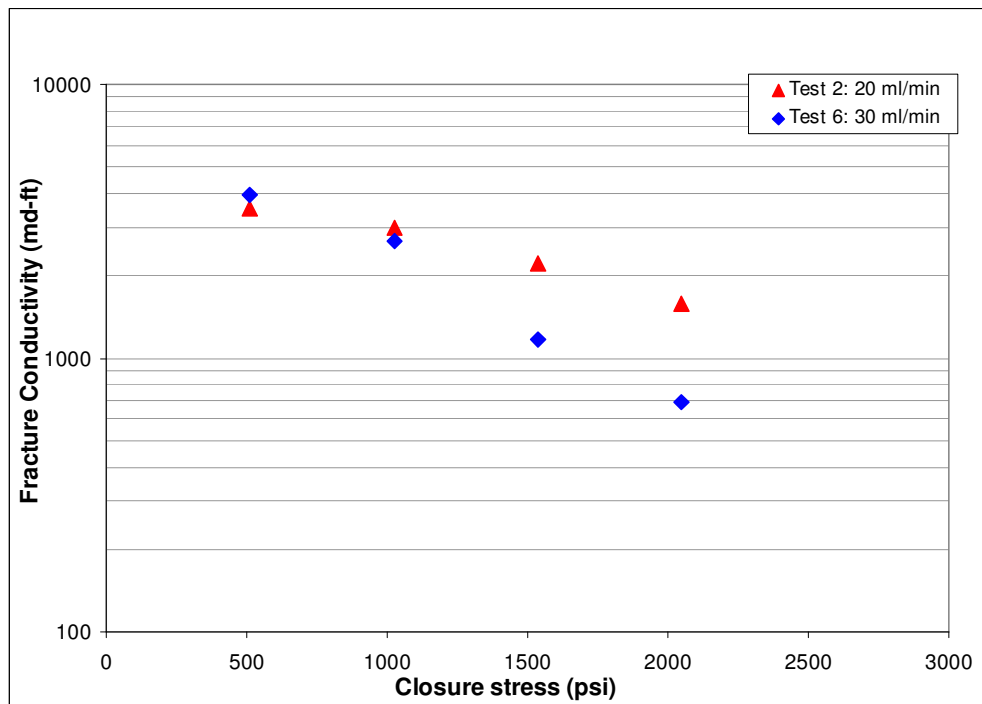


Fig. 3.3—Effect of acid injection rate on fracture conductivity for 10-mins contact time at two different injection rates

Fig. 3.3 shows two experimental results at 20 and 30 ml/min injection rate at same 10-mins contact time. It is obviously shown that higher injection rate decreased more fracture conductivity when closure stresses increased.

From **Fig. 3.2** and **Fig. 3.3**, they provide an evidence of the effect of acid contact time and acid injection rate. Higher acid injection rate and the longer acid exposure time cause in fracture conductivity reduction.

3.2 Effect of Acid Injection Volume on Final Conductivity

The acid injection volume has a direct influence on sustaining fracture conductivity when closure stress increases.

As shown in **Fig. 3.4**, the partial monolayer proppant fracturing without acid injection provided more fracture conductivity than after applied closed fracture acidizing technique at each different acid injection volume. In addition, the least amount of acid injection volume (200-ml case) can maintain the fracture conductivity when closure stress increases better than 300-ml and 400-ml experiments. These effects can be identified from the trend and slope change in fracture conductivity of each test. It appears that when the acid injection volume increases, the fracture conductivity drops more rapidly.

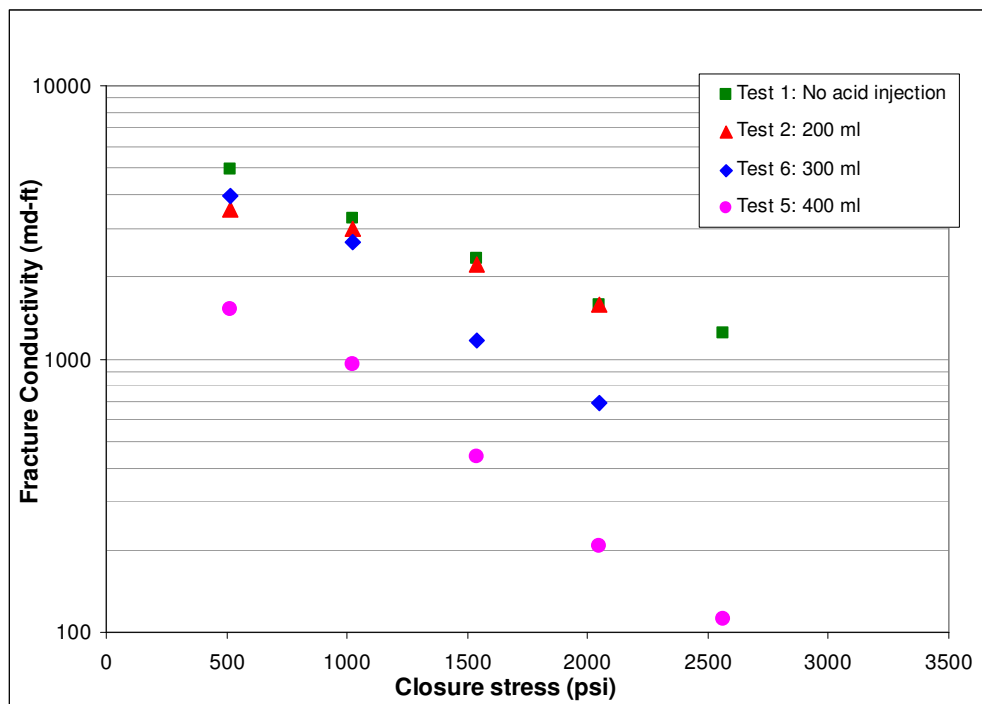
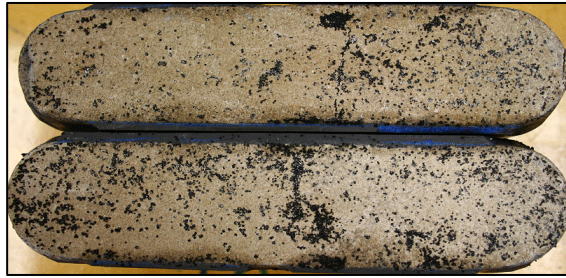


Fig. 3.4—Effect of acid injection volume on fracture conductivity

Fig. 3.5 presents the fracture face of partial monolayer fracturing with no acid and after acid injection of Indiana limestone core samples at 200, 300 and 400 ml of acid. The pictures also show the residual proppant distribution on the fracture surface after acid injection.

The pictures indicate that more acid volume was injected in the fracture; less amount of proppant was left on the fracture face. More amount of rock dissolved would allow propping agents moving easier because proppant did not protect the covered area on the fracture face from acid reaction. In addition, it is noticed during the experiment that more proppant flowing through the outlet line during the acid injection when increasing acid injection volume, although we applied 500 psi closure stress on fracture face during acid injection.

From **Fig. 3.4** and **Fig. 3.5**, it is obviously shown that the closed fracture acidizing process changes the proppant distribution pattern on the fracture face in which decreasing in the original fracture conductivity of the proppant partial monolayer.



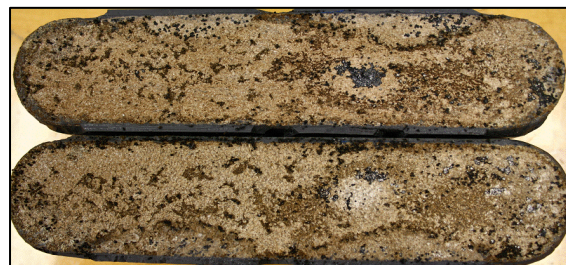
**A) Test 1: Partial monolayer proppant,
without acid injection**



B) Test 2: 200 ml acid injection



C) Test 6: 300 ml acid injection



D) Test 5: 400 ml acid injection

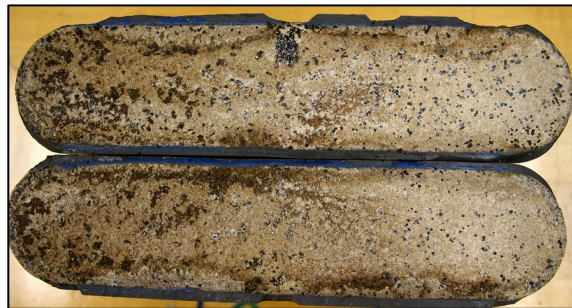
Fig. 3.5—Photographs of core samples after acidizing at each acid injection volume

3.3 Effect of Acid Contact Time on Final Conductivity

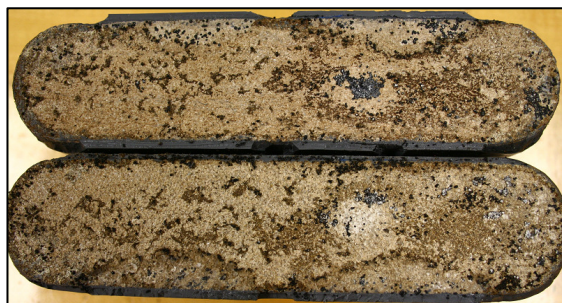
Fig. 3.6 presents the fracture face after acidizing of Indiana limestone core samples at 20 ml/min injection rate at different contact time.



A) Test 3: 10-min contact time



B) Test 4: 15-min contact time



C) Test 5: 20-min contact time

Fig. 3.6—Photographs of core samples after acidizing at each acid contact time of 20 ml/min acid injection rate experiment

The acid contact time is a critical parameter that decreases the original fracture conductivity as shown in section 3.1.2. More acid exposure time caused more reduction in fracture conductivity value. Because the rock surface is weakening after exposure to acid solutions, the proppant grains would be easily embedded inside the fracture face. The proppant embedment problem becomes more pronounced when acid contact time increases, and it is a critical factor in fracture conductivity reduction especially at high closure stress conditions.

3.4 Fracture Conductivity Comparison with Acid Fracturing

The acid fracturing is a typical stimulation technique to apply in carbonate reservoir to improve recovery efficiency. We compared our experimental results with acid fracturing (Melendez *et al* 2007) which used same acid system, gelled acid. **Fig. 3.7** presents the fracture conductivity comparison of partial monolayer proppant fracturing, the closed fracture acidizing at 20 ml/min injection rate, and acid fracturing at 1 L/min injection rate. In **Fig. 3.7**, acid contact time of acidizing and acid fracturing is 10 minutes. The matrix conductivity is presented as a dashed line. The conductivity at the matrix flow indicates that the fracture is completely closed.

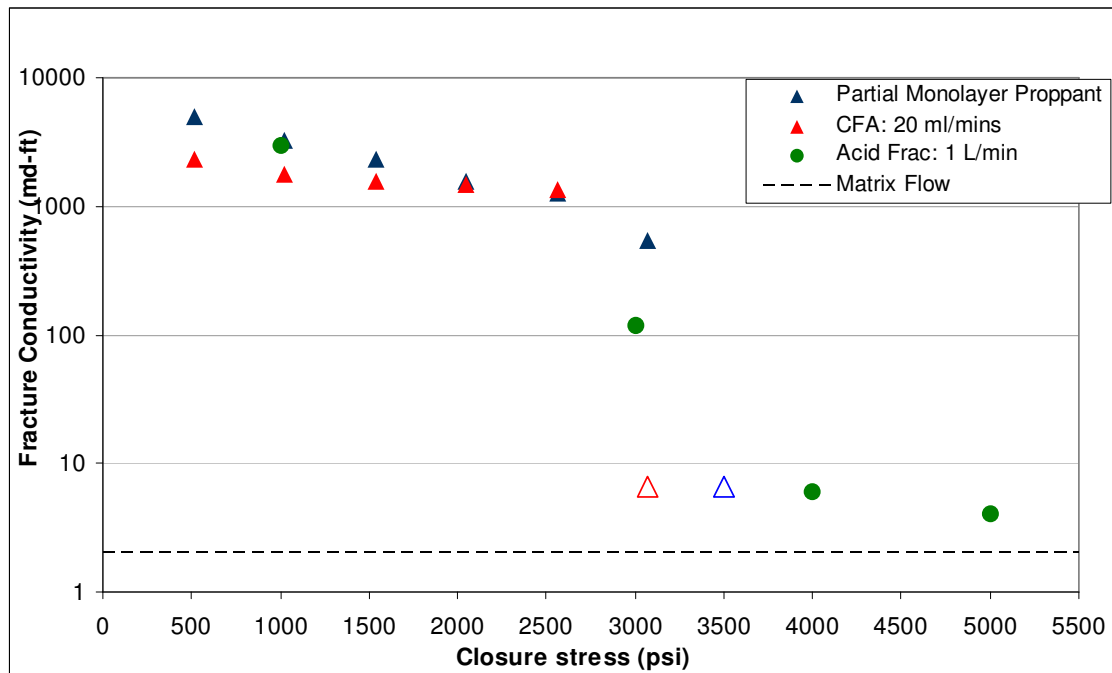


Fig. 3.7—Fracture conductivity comparison of partial monolayer proppant, closed fracture acidizing, and acid fracturing at 10 minutes contact time

From the comparison, it shows that the fracture conductivity from partial monolayer proppant fracturing, closed fracture acidizing and acid fracturing are not much different. The closed fracture acidizing in this 10-mins contact time experiment can sustain the fracture conductivity as good as the partial monolayer proppant because the rock does not dissolve and soften so much that the proppant embedment problem can cause much fracture conductivity reduction. When closure stress is higher than 3,000 psi, the pressure drop across the fracture during the fracture conductivity measurement of both the partial monolayer proppant and the closed fracture acidizing cases are higher than the limit of the pressure transducer which is 20 psi. From the calculation, the fracture conductivity of the 20-psi pressure drop is about 6 md-ft as

shown in the red and blue opened triangle on **Fig. 3.7**. Because the pressure drop in both proppant partial monolayer proppant and closed fracture acidizing are higher than 20 psi, the fracture conductivity for both cases should be less than 6 md-ft at 3500 psi for proppant partial monolayer proppant and 3000 psi for closed fracture acidizing. At high closure stresses, the fracture conductivity of all three techniques dropped dramatically.

The comparison of fracture conductivity for partial monolayer proppant, 15-min contact time for both closed fracture acidizing and acid fracturing is shown in **Fig. 3.8**.

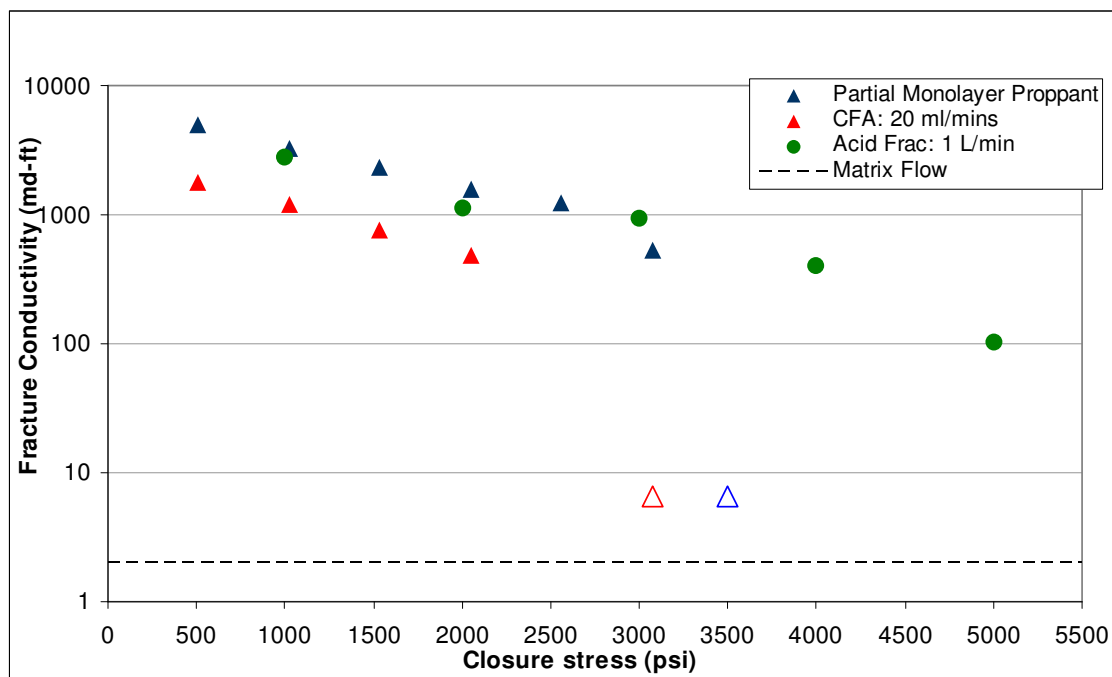


Fig. 3.8—Fracture conductivity comparison of partial monolayer proppant, closed fracture acidizing, and acid fracturing at 15 minutes contact time

At closure stress below 3,000 psi, the conductivity of partial monolayer proppant and acid fracturing are much higher than the conductivity of closed fracture acidizing case. For high closure stress, acid fracturing technique can help sustain the fracture conductivity while the fractures were completely closed in the proppant partial monolayer and the closed fracture acidizing cases resulted in rapid reduction of the fracture conductivity.

We compared the fracture conductivity of partial monolayer proppant and 20-mins contact time experiment for closed fracture acidizing and acid fracturing in **Fig. 3.9**.

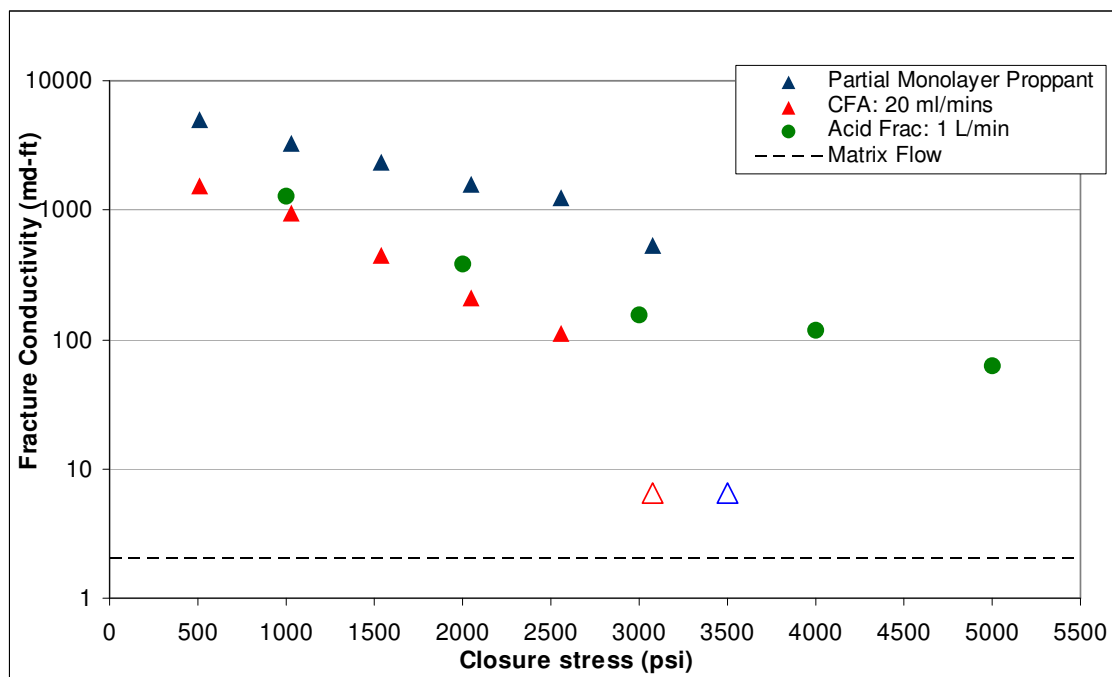


Fig. 3.9—Fracture conductivity comparison of partial monolayer proppant, closes fracture acidizing, and acid fracturing at 20 minutes contact time

It is obviously shown that the partial monolayer proppant provided more fracture conductivity than other techniques at low closure stress. In addition, the least fracture conductivity was obtained from closed fracture acidizing experiment. However, at closure stress above 3,000 psi, the conductivity from acid fracturing can be sustained better than the fracture conductivity from partial monolayer and closed fracture acidizing.

From **Fig. 3.7** to **Fig. 3.9**, the results suggest that the partial monolayer proppant fracturing provided better fracture conductivity than other techniques at low closure stress conditions. Once the closure stress increases, the proppant crushing and embedment problems become more pronounced and lead to decrease the conductivity. The closed fracture acidizing experiments provided the least fracture conductivity values comparing to other techniques in any closure stress conditions. All comparisons show that acid fracture technique has more potential to sustain fracture conductivity at high closure stresses. The longer contact times can yield better conductivity in acid fracturing technique unlike the closed fracture acidizing method. Channel tends to be created in acid fracturing. The channels in this case dominate the conductivity behavior after closure. Since channels are more difficult to crush compared with partial monolayer proppant in proppant fracturing, the fracture created by acid fracturing can sustain fracture conductivity better than fracture obtained by partial monolayer proppant fracturing.

3.5 Comparison with Previous Study

Fredrickson (1986) and Bartko *et al* (1992) performed experimental studies to investigate the performance of the closed fracture acidizing technique (CFA). They simulated a fracture created by acid fracturing technique and inject the acid through a closed fracture with various acid systems. Their laboratory results show in **Table 3.3** and **Table 3.4**.

TABLE 3.3—EXPERIMENTAL RESULTS ON THE CLOSED FRACTURE ACIDIZING (FREDRICKSON 1986)

Test Result No.	Sample Location	Acid Type	Closure stress (psi)	Conductivity (md-ft)	
				Before CFA (After acid fracturing)	After CFA
1	Venezuela	Gelled Acid	7,500	636	171,716
2	Middle East	Emulsified Acid	1,500	204	93,020
3	Michigan	HCl	1,340	7,412	280,000
4	West Texas	Gelled Acid	3,472	<10	<10
5	West Texas	Foamed HCl	1,886	2,360	134,400
6	France	Gelled Acid	2,670	280	186,440
7	France	Gelled Acid	3,594	1,012	181,500

TABLE 3.4—EXPERIMENTAL RESULTS ON CLOSED FRACTURE ACIDIZING (BARTKO *et al* 1992)

Test Result No.	Carbonate Rock Type	Acid Type	Closure stress (psi)	Conductivity (md-ft)	
				Before CFA (After acid fracturing)	After CFA
1	Limestone	Gelled Acid	4,000	< 200	124,000
2	Limestone	Gelled Acid	4,000	Very Poor	680,000
3	Dolomite	Emulsified Acid	4,000	Very Poor	10,000

From **Table 3.3** and **Table 3.4**, they show excellent improvement on fracture conductivity after injected acid through a closed fracture created by acid fracturing. The final fracture conductivity was improved substantially after applied the closed fracture acidizing technique. The closed fracture acidizing technique can also help to sustain fracture conductivity at high closure stress as shown in test result 1 and 2. It is most likely that most of soft material on fracture face is dissolved during acid fracturing process. When pumped acid through a closed fracture as in closed fracture acidizing process, a small part of the overall fracture face has been dissolved into a relatively deep channel. As a result, the remaining unetched surface can hold this channel open under high closure stress condition.

In our study, the closed fracture acidizing technique for a created fracture by proppant partial monolayer fracturing was focused. The fracture conductivity of partial monolayer proppant in our study is lessened when pumped the gelled acid through a propped fracture as shown in section 3.1 and 3.2. With too long acid contact time or more acid injection volume, the conductivity reduction is more severe. Because the area covered by proppant also reacted with acid, the fracture face became soft or weak after contact with acid in which leaded proppant to be easier to embed into the fracture face. In this case, there were not sufficient grooves that could hold fracture open to provide a high conductive pathway for fluid to flow, resulting in decrease in fracture conductivity dramatically.

CHAPTER IV

CONCLUSIONS AND RECOMMENDATIONS

4.1 Conclusions

A series of experiments were conducted to determine the effect of acidizing treatment on fracture conductivity in a closed fracture with partial monolayer proppant placement . The conclusions based on this study are:

1. Acid does not improve the final fracture conductivity of a partial monolayer proppant fracturing. Acid reduces an original conductivity of the propped fracture. More acid injection and too long of an acid exposure time decrease more final conductivity because of weakening of the rock surface in which the proppant can be easily embedded on the fracture face. This effect become more critical for higher closure stresses.
2. More amount of rock dissolves when increasing acid injection volume and acid contact time. In this situation, proppant particles move easier resulted in more losing propping agents during acidizing process in which the fracture conductivity decreases substantially.
3. Acid fracturing technique can yield more fracture conductivity than other techniques at high closure stresses. Longer contact time in acid fracturing leads to provide better fracture conductivity result.

4.2 Recommendations

We have been successful conducting partial monolayer fracturing and closed fracture acidizing in the laboratory. Although the failure to improve fracture conductivity from the closed fracture acidizing in proppant partial monolayer fracturing is shown, the closed fracture acidizing technique should still be further investigated for other types of created fracture. To evaluate this technique, extensive experimental study should be performed.

In addition, additional extensive experiments should be carried out in other types of carbonate rock. Because rock properties of each types of carbonate rock are different, the closed fracture acidizing technique might help increase final fracture conductivity for other types of carbonate reservoir resulted in recovery efficiency improvement.

Finally, these experiments provide better understanding of closed fracture acidizing in proppant partial monolayer fracturing. We carefully quantified the fracture conductivity behavior under different acidizing conditions. However, additional extensive experiments are recommended to properly evaluate this closed fracture acidizing technique on other types of created fracture and carbonate rock.

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APPENDIX

Side A



Side B

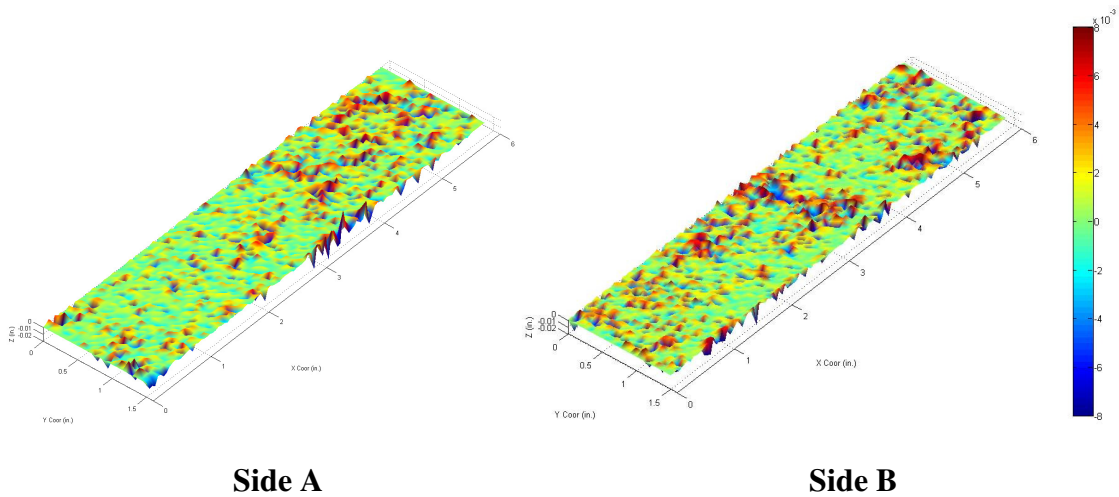


Fig. A.1—Experimental data for Test 1 – partial monolayer proppant fracturing, no acidizing

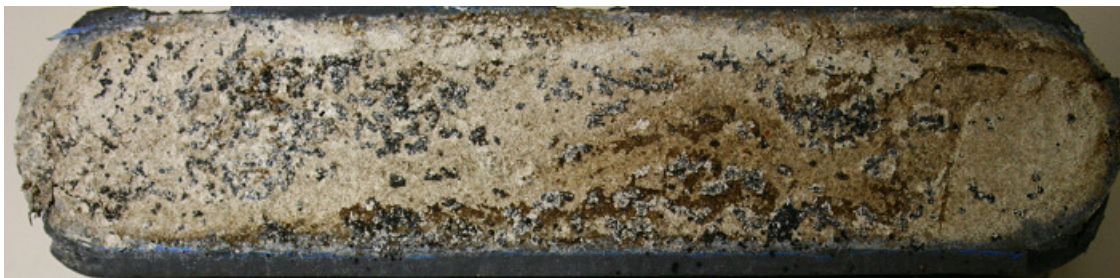
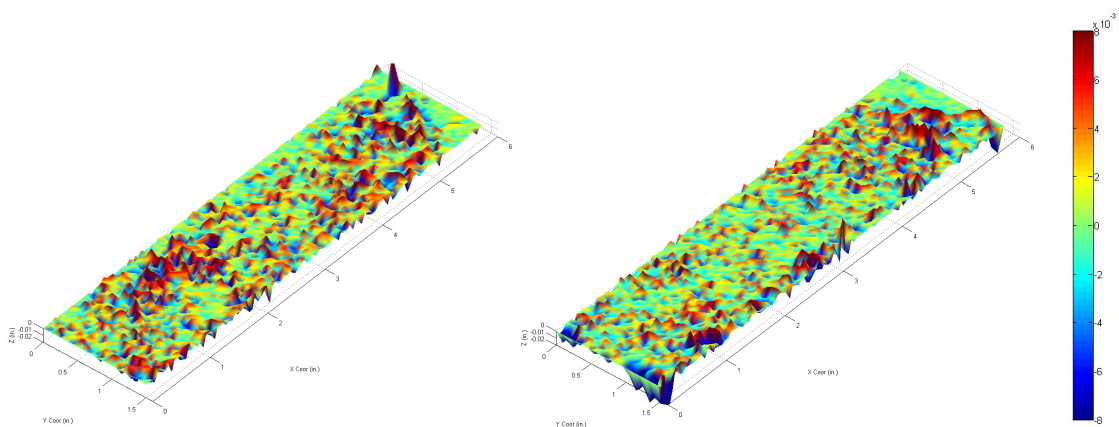
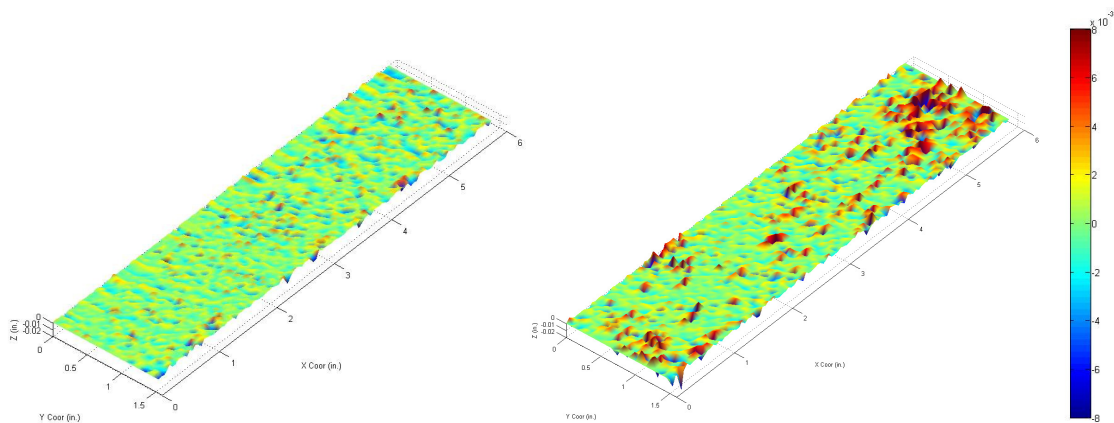
Side A**Side B****Side A****Side B**

Fig. A.2—Experimental data for Test 2 – partial monolayer proppant fracturing with 20 ml/min for 10 minutes acidizing

Side A



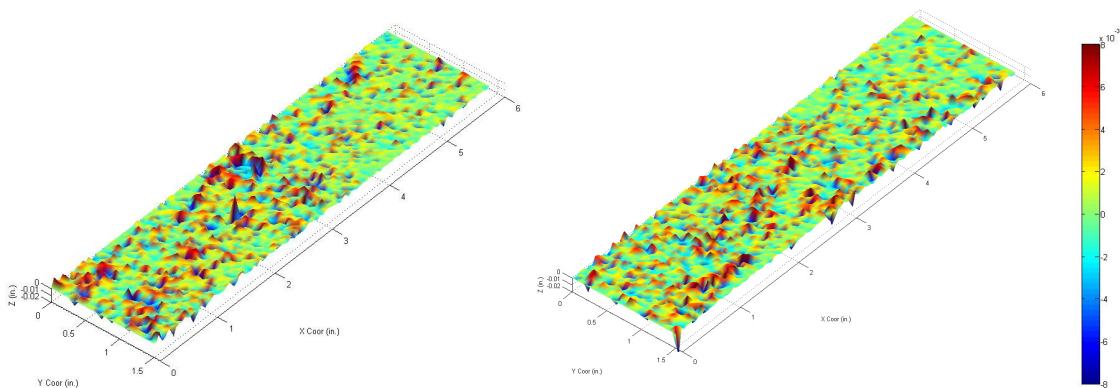
Side B



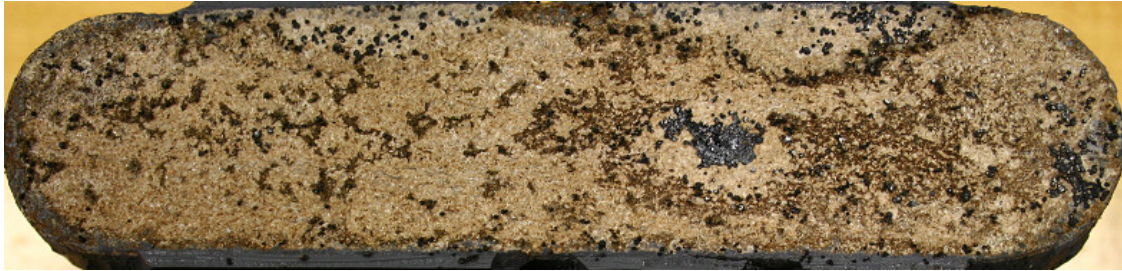
Side A

Side B

Fig. A.3—Experimental data for Test 3 – partial monolayer proppant fracturing with 20 ml/min for 10 minutes acidizing

Side A**Side B****Side A****Side B****Fig. A.4—Experimental data for Test 4 – partial monolayer proppant fracturing with 20 ml/min for 15 minutes acidizing**

Side A



Side B

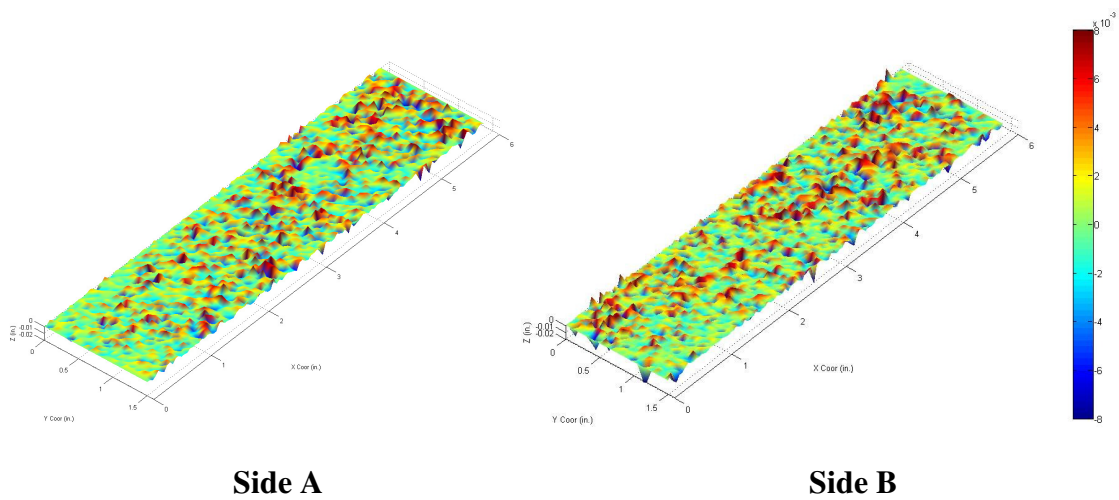


Fig. A.5—Experimental data for Test 5 – partial monolayer proppant fracturing with 20 ml/min for 20 minutes acidizing

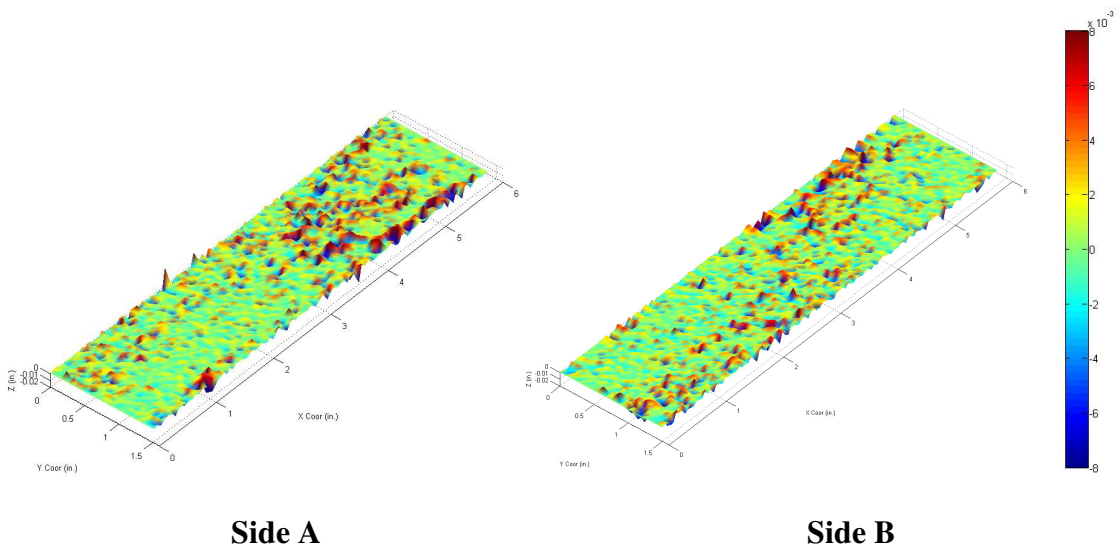
Side A**Side B**

Fig. A.6—Experimental data for Test 6 – partial monolayer proppant fracturing with 30 ml/min for 10 minutes acidizing

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