# FREEZING OF POORLY CONSOLIDATED ROCK: METHOD OF STABILIZATION OR ALTERATION?

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## ABSTRACT

Core freezing is one of the most popular methods for preservation of poorly consolidated core before routine and special core analysis procedures. The freezing technique is generally recommended for unconsolidated formations to maintain core integrity during handling. As to practical use of results obtained on core samples subjected to freezing-melting cycle it appears to be extremely hard to predict in advance which properties will be affected and what the degree of alteration will be.

We performed a special study to investigate alteration of properties of poorly consolidated core samples that occur as a result of quick freezing in liquid nitrogen. Special core analysis of more than 60 poorly consolidated cylindrical core plugs of Suzunskoye oilfield has been conducted before and after quick freezing in liquid nitrogen. A number of petrophysical properties were measured before and after freezing: porosity, permeability, capillary pressure, residual water, acoustic, electrical resistivity, NMR response, specific surface area and elastic properties. For the studied core samples, we observed minimal variation of fluid transport properties and, for some cores, significant alteration of elastic properties.

## CORE PREPARATION AND EXPERIMENTAL PROCEDURE

Three companion groups of core plugs were involved in this study. The first group included 30 cleaned and dried cores which were used for RCA and SCAL measurements. The second group was 30 pairs of non-extracted cores for geomechanical tests. A third group of small non-extracted plugs were cut for micro-CT analysis. Cores in each triplet were drilled from the same full-size core and had similar properties. RCA, SCAL measurements and micro-CT scanning were performed on cores before freezing on dry cores, then the cores were fully saturated with brine and capillary pressure curves were obtained. In order to reproduce the natural state of a sample during core freezing, we saturated samples again with brine up to $S_w$~35% before freezing. Then the cores were put in liquid nitrogen (-196$^o$ C) for one day. For the next step, samples were left for 24 hours at room conditions and then they were dried and used for physical properties measurements as well as for micro-CT scanning.

## RESULTS

### Petrographic image analysis of thin sections

Two sets of thin sections were investigated: the first set was prepared from cylinders in natural state; and the second set from plugs frozen in liquid nitrogen. Studied rocks have poorly-rounded grains with frequent inclusions of grains having acute shapes. Contacts between grains occur as points and are poorly incorporated (Fig. 1). For the studied rock, the assumed alteration of pore space structure in the results of freezing-melting cycle should cause failure of point and poorly incorporated contacts between grains as well as opening of the slot-like pore throats. In the result of numerous studies such defects haven’t been found. However, image analysis of pore space structure using thin sections showed that the majority of samples that passed through freezing-melting cycle have less pore space than their counterparts prepared without freezing (Fig. 2).

*Fig.1 Conformal contacts between grains.*

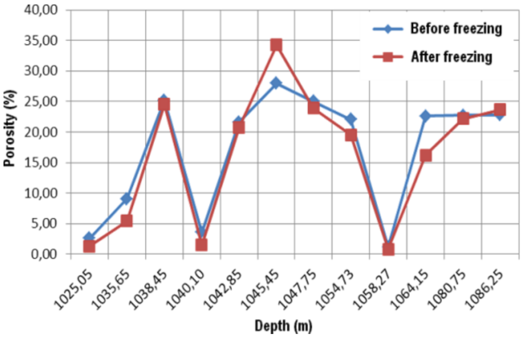

*Fig.2. Comparison of pore space volume for samples before and after freezing*

### NMR measurements before and after freezing

An NMR study was performed on cores before and after freezing at both ambient and elevated temperature and pressure (PT) conditions. As it can be seen from Figure 3, transverse relaxation time distributions (T2) measured at ambient conditions before and after freezing are in good agreement especially for reservoir rock (Fig. 3A). Thus we can conclude there were insignificant changes due to freezing for this sample. In some cases we observed the disappearance of part of the T2 spectrum at long relaxation times (Fig.3B). We can explain this peculiarity with technology of core samples preparation. Special confining sleeves could form some leaks around sample edges which add a long components into NMR distribution especially for unfrozen cores. Specimens after freezing expanded a little due to thermal expansion and bound the sleeves more strongly to the core and without leaks in most cases. Ten cores were also studied at high pressure and temperature (Fig. 3C and 3D). After applying confining pressure we observed decreasing of pore volume due to compression of large pores both before and after freezing. Thus, any fractures in the samples with high clay content (Fig. 3C) and various artificial cracks and pores were closed at confining pressure and we think that the most reliable tests should be done at elevated confining pressure.

### Reservoir properties

Numerous measurements of gas permeability, porosity (by water, gas and NMR), electrical resistivity, acoustic wave velocity and residual water saturation (by centrifuge) were performed on the same cores before and after freezing.

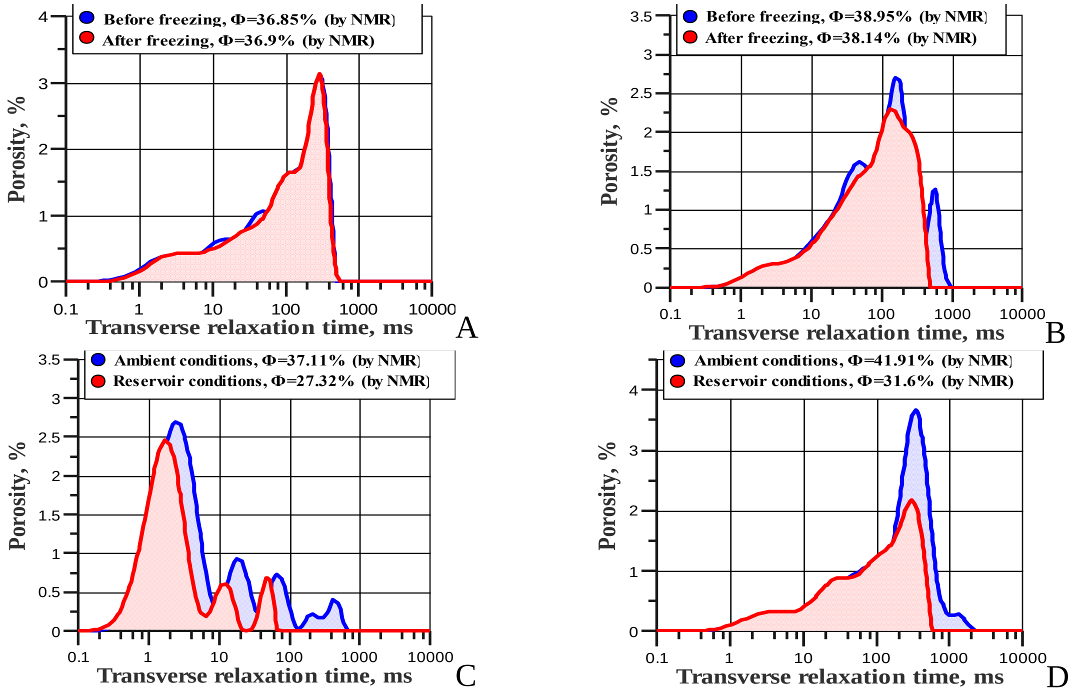

*Figure 3. Transverse relaxation time distribution measured before and after freezing at ambient (A and B) and at high pressure-temperature (C and D) conditions.*

As shown in figure 4, significant increase of porosity (up to 5.4%) and permeability (up to 3 times) values are observed for rock with high clay content. A possible explanation is clay swelling during the saturation and freezing cycle. Pure quartz sandstone with high porosity and permeability had smaller alterations but they occur in both directions. Possible reason of such deviation can be slight mechanical disintegration during measurements for some cores (application of confining pressure, brine saturation, centrifuge etc.) and following compaction due to application of confining or axial pressure during the measurements.

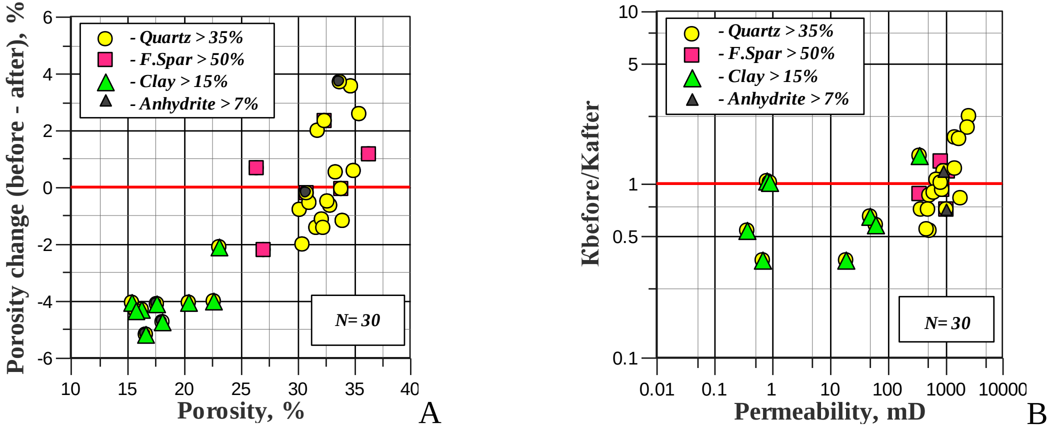

*Figure 4. Porosity (A) and permeability (B) changes after freezing (differentiation by XRD).*

Alteration of formation factor did not depend on clay content (Fig. 5A) and the average value of the alteration was about zero. Another observed feature of rock with high clay content is higher density and elastic wave velocities (Fig. 5B). These rocks became slower after freezing up to 5% because of clay cement swelling.

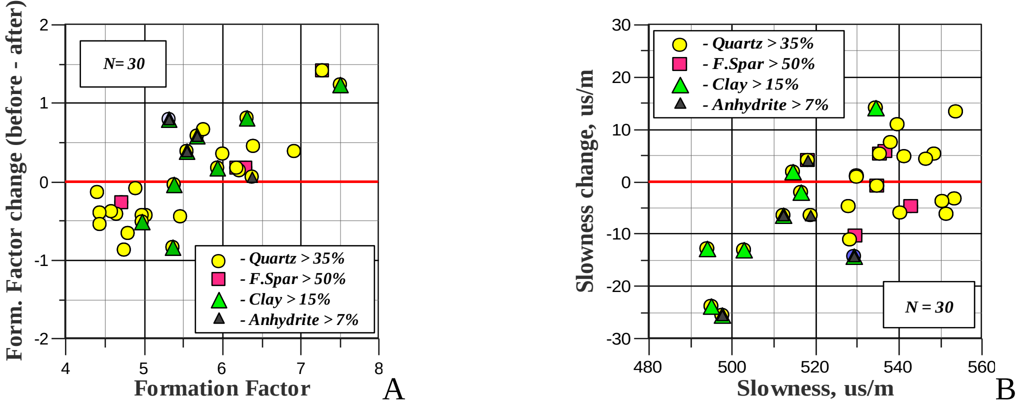

*Figure 5. Formation factor (A) and slowness (B) changes after freezing (differentiation by XRD).*

## Geomechanical properties alteration

Triaxial tests for pairs of sandstone plugs were performed. One plug from each pair was frozen and then unfrozen before testing. On Figure 6, a comparison of stress-strain curves for one pair is presented that shows an extreme case of the difference. For the unfrozen plug, the behaviour is clear elastic - ideally plastic deformation; for the companion frozen plug the behaviour has only a small elastic part followed by plastic deformation. The second plug looks like it failed before the mechanical test began. This behavior is not typical for all plugs. In Table 1, comparisons between frozen and unfrozen cores of the differential pressure at the yield point and of Young's modulus are presented for some pairs. It is clear that for half of the pairs, the yield pressure for plugs after freezing-unfreezing cycle is greater than for plugs without freezing. In addition, a difference of 1-2 MPa between yield points for pairs of plugs can be explained because plug pairs are not identical, but a difference of 10-15 MPa can be a consequence of rock failure. As a conclusion it is evident that core freezing can lead to rock failure and incorrect mechanical properties measurements but it is not true that freezing will break the rock in all cases.

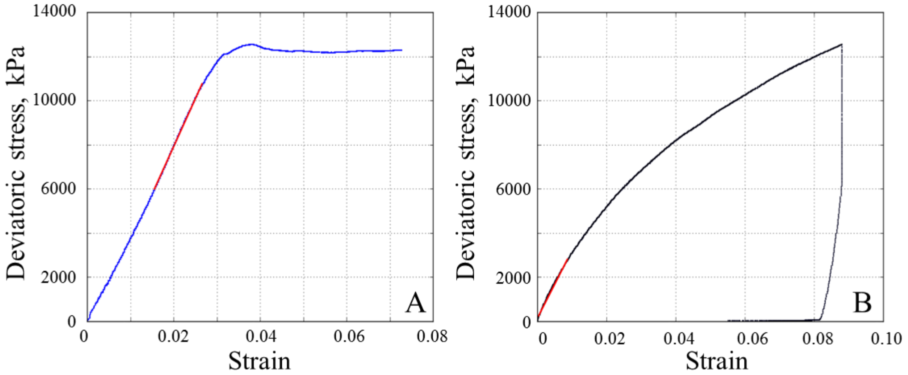

*Figure 6. Comparison of stress-strain curves for two plugs from one depth and same confinement pressure before (A) and after (B) freezing.*

*Table 1. Comparisons of differential pressure of yield points and of Young's modulus for plug pairs.*

| Confinement pressure MPa | Yield point's differential pressure MPa (plug without freezing) | Yield point's differential pressure MPa (plug after freezing) | Young's modulus MPa (plug without freezing) | Young's modulus MPa (plug after freezing) |
|---|---|---|---|---|
| 1 | 2.5 | 1.5 | 92 | 127 |
| 7 | 11.0 | 3.0 | 432 | 307 |
| 3 | 1.5 | 4.0 | 210 | 168 |
| 5 | 4.5 | 2.0 | 266 | 302 |
| 13 | 3.0 | 5.0 | 540 | 505 |
| 13 | 3.0 | 18.0 | 540 | 840 |
| 13 | 18.5 | 5.5 | 1120 | 472 |

## Micro-CT scanning results

Ten core samples that showed significant changes of mechanical properties were selected for an SMR workflow on advanced characterization of pore structure by X-ray micro-CT. Micro-CT images were achieved for the same portion of cylindrical rock samples (8 mm x 4 mm) before and after freezing. Then a comparative study of the resulting micro-CT images from "initial" and "after freezing" states were performed by the specialized in-house software developed at Schlumberger Moscow Research. Its operation principle is based on minimization of an arbitrary functional provided by varying seven affine transformation parameters (three for center of mass movement along 3D axes, three for rotation about the same axes and one "scale factor"). In Figure 7C we use the absolute value of gray levels difference to highlight altered regions.

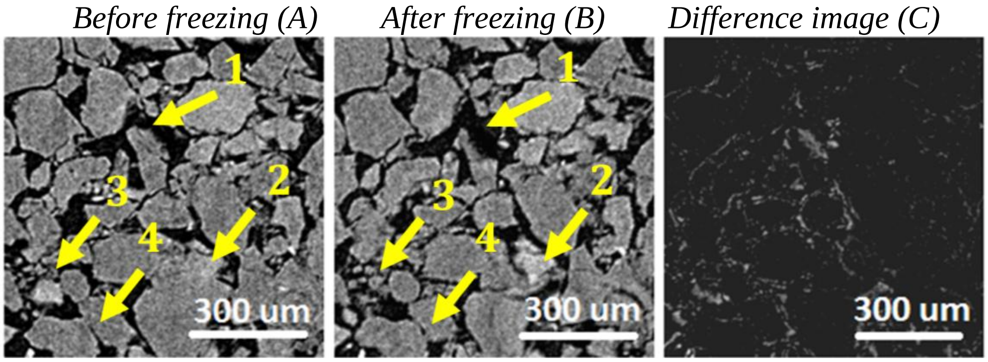
*Before freezing (A)* *After freezing (B)* *Difference image (C)*

*Figure 7. Typical structural changes of clayey sandstone: 1 – grain splitting, 2 – fracturing, 3 – particles migration, 4 – intergranular contact change*

"Black" areas on Figure 7C represent undisturbed parts of the sample and white brighter regions indicate that something had changed. The most significant effect of freezing on rock structure was observed for clayey sandstones. Typical changes of rock structure are shown on registered 3D images (Fig. 7): (1) grain splitting; (2) appearance of fractures; (3) spatial migration of particles less 40 um in size and (4) intergranular contact change. An average distance for small particles (less 40 um) movement was about 2–3 linear sizes of the particles. Appearance of highlighted outlines in the difference images revealed changes of grain boundaries and their slight movement as a result of sample freezing-melting cycle (Fig. 7C). Analysis of 3D registered images shows that particle movement occurred in the plane of 2D images as well as perpendicular to them. It was identified by appearance and disappearance of rock particles for images after treatment. Poorly consolidated and/or fine-grained sandstones proved to be the most sensitive to freezing-melting: Figure 8 illustrates the formation of large fractures and propagation of existing ones.

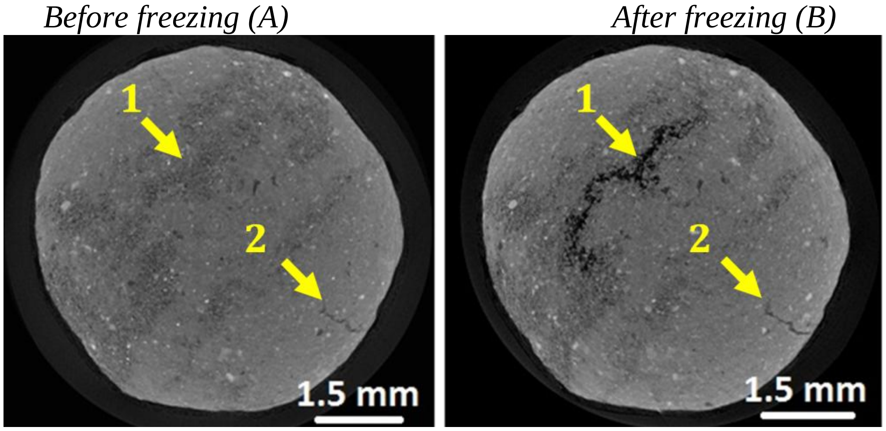

*Figure 8. Structural changes typical for poorly consolidated/ fine-grained sandstones: 1 — large fractures and 2 — propagation of existing fractures*

## SUMMARY AND CONCLUSIONS

In this study we applied RCA and SCAL tools to reveal rock structural changes by influence of freezing-melting cycle. Significance of a freezing effect depends on lithology, mineral composition, pore structure, freezing procedure and water saturation. Micro-CT studies of a freezing effect on rock structure alteration revealed common types of changes within intergranular contacts, particle movement, grain disintegration and growth of new fractures. For the studied core samples, such structural changes had minimal effect on fluid transport properties and, at the same time, elastic properties can be affected significantly.

It should be noted that a poorly cemented core also can be broken without freezing. In addition, for some cases core freezing is the only possible method for plug preparation. In these cases for proven results the main recommendation is to obtain X-ray micro-CT of several representative plugs or core particles before and after freezing. Also when these results are used for geomechanics study, some uncertainty analysis will be useful.

## ACKNOWLEDGEMENTS

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