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Magnetic resonance imaging of reinforced ice and ice-based composites

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ABSTRACT

Effective exploitation of the Arctic region largely depends on the availability of materials, which are necessary for polar engineering facilities operating in severe climatic conditions. The most cheap and abundant material available in cold regions is natural ice. However, its mechanical properties are highly temperature-dependent that requires development of methods for ice reinforcement. Strong requirements applied to reinforced ice and ice-based composites for Arctic regions cannot be satisfied without comprehensive study of ice structure and properties both at microscopic and macroscopic levels using various experimental methods. This paper presents numerous results of Magnetic Resonance Imaging (MRI) of ice and its composites with the aim to systematize different study areas associated with issues of ice freezing and thawing in the Arctic region. Brief literature review describes the main achievements of MRI in this field, advantages and limitations of the method in respect to ice research, and new problems and challenges for the ice science. Some of the presented examples of experimental research describe the main applications of MRI to normal ice, salt-water ice, reinforced ice, and ice-based composites. The results obtained demonstrate the advantages of MRI method in the study of ice composite materials, which are promising for Arctic regions. New data on the structure and properties of ice composites effectively complement the results of classical mechanical tests of such materials for the purpose of their comprehensive and complex study for further practical application.

KEYWORDS: Reinforced ice; MRI; Freezing; Thawing; Melting front

INTRODUCTION

The Arctic region draws all the more attention due to its growing economic importance. However its harsh environment imposes severe restrictions to polar research and exploitation. One of the problems associated with remote polar environment is the lack of cheap, easily processable and transportable construction materials with a necessary mechanical and thermal stability. Natural ice abundantly presented in cold regions can be considered as promising building material. However, a significant disadvantage of ice as a construction material is its creep behavior at temperatures around 0°C. As a result, its mechanical properties need to be improved and ice tendency to creeping must be diminished. The behavior of ice can be sufficiently improved by reinforcement and creating the ice-based composites (Vasiliev, et al., 2015). A lot of known reinforcing approaches allow increasing the strength of ice composites, their durability, and thermal stability (Nixon, 1989; Vasiliev, 1993; Vasiliev, et al., 2012; Vasiliev, et al., 2015; Buznik, et al., 2019).

The properties of materials under extreme temperature and humidity conditions must be studied for the accurate prediction and optimum design of marine structures, polar ships, buildings, roads, and other engineering facilities. Strong requirements applied to reinforced ice and ice-based composites for Arctic regions cannot be satisfied without comprehensive study of ice structure and properties both at microscopic and macroscopic levels using various experimental methods such as microscopy, mechanical, and thermal testing etc. Mechanical testing is widely accepted as a key experimental technique, which provides necessary information about strength and other mechanical properties of the ice (Buznik, et al., 2017). Evidently, the mechanical testing does not allow assessing all the ice characteristics and complementary experimental techniques are now actively introduced into this study area. One of the methods used in the ice study is Magnetic Resonance Imaging (MRI). This method allow for precise assessment of physical state of the water absorbed by material or structures frozen into the ice providing qualitative visualization of water migrations and information on water distribution within the material, water freezing and ice thawing, as well as quantitative assessment of these processes. MRI method has already found wide application as nondestructive and very informative tool in medical diagnostics and numerous biomedical areas of research as well as in materials science (Blumich B., 2003). Since the early 1990s the number of works devoted to the study of ice with MRI has been growing but we are still lacking systematic work on application of MRI to reinforced ice and ice-based composites. In this paper we made an attempt to briefly systemize the results available in the literature and to present the new results of MRI used in the study of reinforced ice and processes of ice freezing and thawing. Beginning with normal and salt-water ice, we demonstrate the feasibility of using MRI in the study of freezing and thawing processes in model reinforced ice, ice-based composites, frozen nanodispersed suspensions, and hydrogels, since this methods allows obtaining both qualitative and quantitative data. The results obtained give valuable information on the materials and can be used for creation advanced reinforced ice and ice-based composites.

MRI METHOD

The key phenomenon, which primarily determines the advances of MRI method application to the study material, is a Nuclear Magnetic Resonance (NMR). NMR is worthily considered as one of the most powerful techniques used in the investigation of condensed matter due to extremely sensitive response of the NMR signal to changes in molecular structure, molecular order, and molecular dynamics in the studied matter. Substantial progress in NMR science occurred when the new (spatial) dimension was introduced by means of application of magnetic field gradients, which underlie the operational principles of the MRI method (Callaghan, 1991). Due to the magnetic field gradients the NMR frequency of resonating nuclei becomes spatially dependent, i.e. the particular position of nucleus in space can now be encoded during the NMR experiment. As a result, MRI allows measuring the intensity of NMR signal, which comes from the selected area of the sample (e.g. slice plane in Fig. 1). Normally this area is represented by the plane referred to as slice or NMR image with a thickness varying from few hundreds of micrometers up to several millimeters. Dimensions of the voxels within the slice are determined by the slice thickness and ratio of field-of-view

(FOV) parameter to the matrix size (e.g. N×M pixels).

The obtained 2D map of the NMR signal intensity distribution strongly correlates with the local relaxation times T_1, T_2, and density of resonating nuclei. Thus, for a commonly used spin-echo pulse sequence (Fig. 1) the NMR signal intensity, I_{se}, is expressed as:

$$I_{SE} \propto \rho\left[1-\exp\left(\frac{TE-TR}{T_1}\right)\right]\exp\left(\frac{-TE}{T_2}\right), \tag{1}$$

where TR (repetition time) and TE (echo time) are the parameters of spin-echo pulse sequence as shown in Fig. 1; ρ is the density of resonating nuclei; T_1 and T_2 are the spin-lattice and spin-spin relaxation times, respectively. As can be seen, varying the timing parameters TR and TE opens the way to contrast enhancement, i.e. the character of signal intensity variation can be changed according to the purpose of the study. Thus, setting $TR \geq 5T_1$ at TR>>TE diminishes the dependence of I_{se} on T_1, yielding either T_2-weighting (at $TE \approx T_2$) or ρ-weighting (at $TE << T_2$) of the images. Inversely, short TR parameter yields the T_1-weighting. As a result, weighted images demonstrate better details associated with specific state of the matter. For example, the relaxation times T_2 of liquid water and ice are dramatically different (few seconds and less than 1 ms, respectively) while the proton density difference between these two states is relatively small. Consequently, the relaxation time weighting of the images provides superior contrast – during freezing/thawing processes the boundary between ice and liquid water can be clearly visualized in any part of the sample.

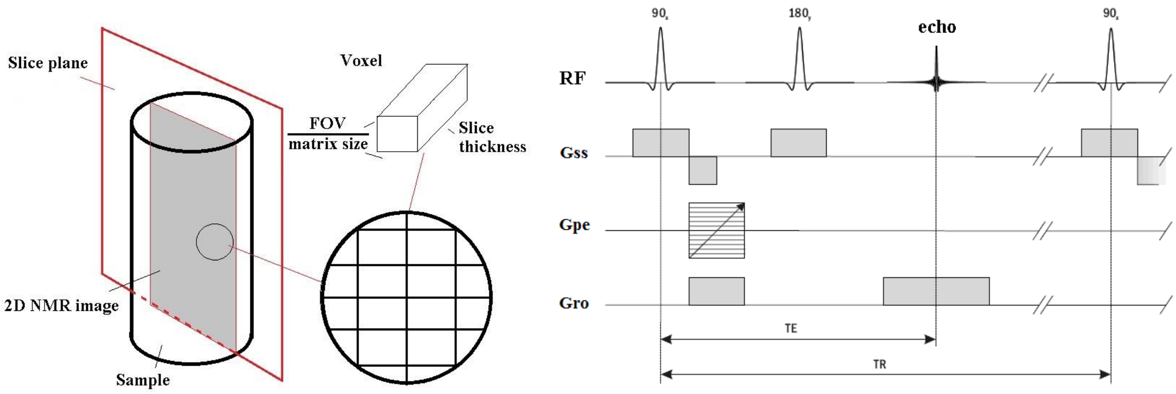

Figure 1. The slice plane, image, and voxels size in MRI (left picture); the basic timing diagram of spin-echo pulse sequence (right picture)

All the MRI experiments on reinforced ice presented in this article were conducted using the Bruker AVANCE DPX 200 spectrometer, equipped with micro-imaging accessories in the following configuration: wide bore, 89-mm diameter, superconducting magnet operating at 4.7 T, water-cooled, and self-shielded Bruker GREAT 3/40 gradient unit (with values of pulsed field gradient strength up to 1 Tm^{-1}), probe PH MICRO 2.5, 25-mm internal diameter birdcage coil tuned and matched to 1H nuclear resonance frequency of 200.13 MHz, and console with Paravision 4.0 software. Slice selective 2D NMR images were acquired using the Multi-Slice Multi-Echo (MSME) technique based on spin-echo pulse sequence supplied by the spectrometer software. The parameters of image acquisition were normally as follows: slice thickness of down to 0.5 mm, FOV of up to 40×40 mm, matrix size of up to 256 × 256 pixels, TR on the order of 100-250 ms, TE of 3 ms, image acquisition time ranging between

25 s and 1 min. Reinforced ice and ice-based composites were frozen and thawed inside the MRI probe using the flow of gaseous nitrogen heated up to the goal temperature ranging from -30°C to +30°C.

RESULTS AND DISCUSSION

Application of NMR techniques to sea-ice investigation was reported as early as the NMR method itself became accessible for researchers (Richardson and Keller, 1966). However, for a long time the ice had been studied using spectroscopic techniques, which did not provide any spatial information. Only decades later, the MRI method was developed and then applied to salt-water ice (Edelstein and Schulson, 1991). Despite the sufficient progress in this study area , ice may be regarded as one of the most difficult substances for 1H imaging – many millions of proton magnetic resonance images of liquid water had been taken before the first report about imaging of normal ice was published (Nunes et al., 2007). The main problem in MRI of normal ice is the loss of the NMR signal caused by a sharp reduction in spin-spin relaxation time T_2 and the large proton line width. As a result, proper visualization of solid ice requires application of special pulse sequences and extremely strong magnetic field gradients. Latter can be implemented by using special stray-field MRI (STRAFI), which provides very high (e.g. 50 Tm^{-1}) static gradients (Landeghem et al., 2011). Nunes et al. obtained their first proton NMR images of solid ice using the STRAFI technique (Nunes et al., 2007).

Since MRI does not provide proper NMR imaging of solid ice, many researchers focused their attention on indirect visualization of ice and snow microstructure. One of the possible approaches is to fill the pore space in ice or snow by inert organic liquids such as aniline or dodecane, which have low melting point, staying in a liquid state (i.e. providing a strong NMR signal) within the pores of solid ice/snow. Thus, MRI can be used for proper visualization of filled samples, elucidating the microstructure, morphology, and pore connectivity inside the ice structure. Following this way the three-dimensional structure of four types of packed ice particles in snowpack was successfully studied (Ozeki et al., 2003).

Another possible source of strong NMR signal in solid ice could be the liquid inclusions of brine – in sea water, freezing of the aqueous phase begins at approximately -2°C while a temperature decrease leads to a separation of freshwater ice and brine and increases salinity in brine-filled pores (Menzel et al., 2000). The inclusions of concentrated brine surrounded by freshwater ice serve as a probe, which provides proper MRI visualization of ice microstructure. Based on this approach, numerous investigations of sea-ice were carried out, including visualization of sea-ice pore fluids and thermal evolution of pore microstructure (Eicken, et al., 2000), MRI of drainage channels in sea spray icing (Ozeki et al., 2005), the effect of growth conditions on the network of brine-filled pores in ice (Menzel et al., 2000), MRI monitoring of sea spray freezing at different freezing regimes (Wilbur et al., 2020), and other studies (Edelstein and Schulson, 1991; Katsushima et al., 2020; Brox et al., 2015; Galley et al., 2015).

The majority of experimental studies of sea-water ice evidently concern the structural effects since the brine content, brine-associated microstructure, and brine drainage channels connectivity all play a crucial role in the formation of mechanical and thermal properties of ice cover. Yet a little attention was actually paid to MRI visualization of freezing and thawing of ice itself. We believe that MRI method is able to provide deeper insight into properties of ice in terms of its thermal behavior during freezing/thawing cycles. For example, MRI clearly differentiates the qualitative character of freezing observed in pure water and NaCl solution. Thus, the images of pure water during the freezing process demonstrate two-stage dynamics – upon reaching the freezing point the water sample almost immediately forms the polycrystalline ice all over the sample volume (faster than the temporal resolution of MRI method), however sufficient amount of water captured within an interparticle space still provides strong NMR signal (see Fig. 2). This evokes the freezing front, which moves forward until complete solidification of the sample. In NaCl solution we observed a distinct picture – the formed polycrystalline ice also captures some brine but without entailing a freezing front (Fig. 2). Instead, slow decrease of NMR signal intensity was observed all over the sample volume and some residual signal persisted long enough due to small amount of liquid brine inclusions.

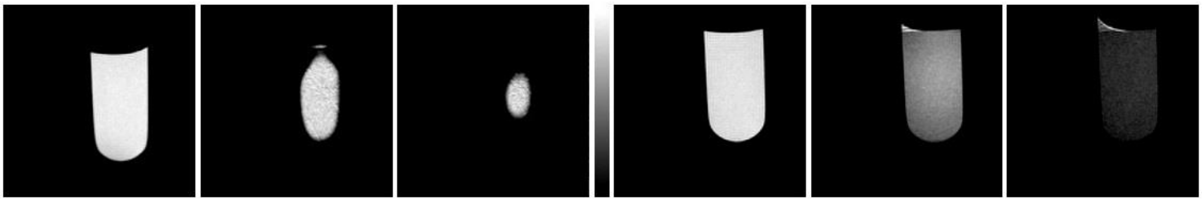

Figure 2. Series of NMR images of pure water (left row) and 10 wt.% solution of NaCl (right row) freezing at -25°C

One of the advantages of MRI measurements of ice during freezing/thawing cycling is the quantifiability of the processes. The signal intensity of NMR images measured during the freezing process is presented in Fig. 3. We see that the curves have a relatively straight region in a middle part that allows measuring the rate of freezing, *k*, expressed in terms of intensity, *I*, divided by time. Comparison of the rates normalized by the temperature of the processes shows that the rate of thawing slightly depends on salinity of the salt-water ice whereas the maximum rate of freezing is approximately 2.5 wt.% (see Fig. 3).

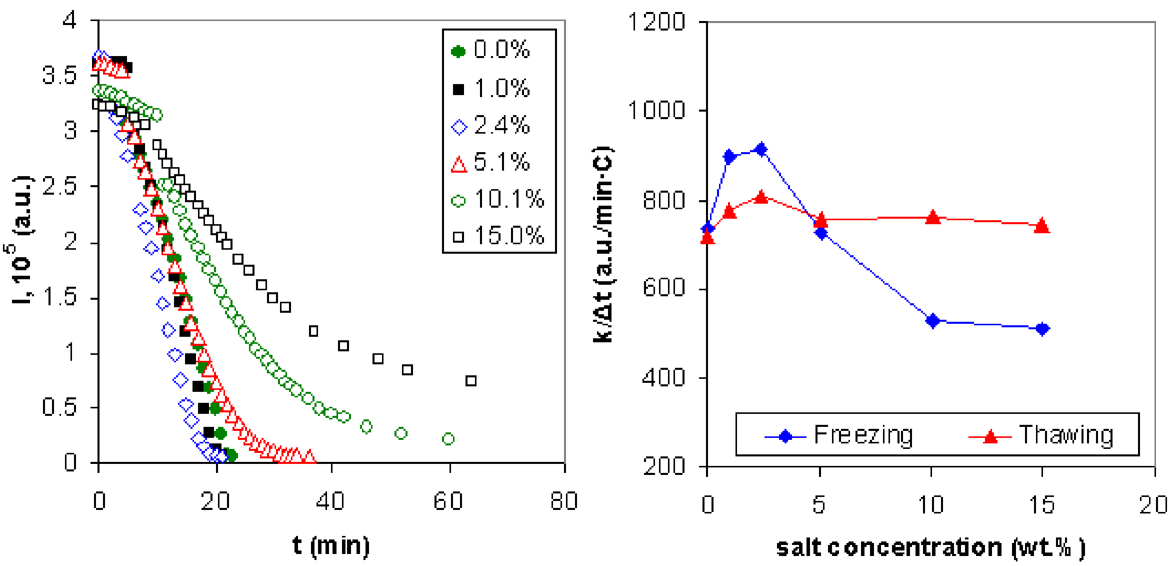

Figure 3. Series of NMR signal intensity curves for samples with different salinity during their freezing (left graph); dependencies of normalized rate of freezing/thawing on salinity of the samples (right graph). Temperatures of freezing and thawing were -25°C and +25°C, respectively

It is noteworthy that not only the bulk ice samples are the objects of MRI study. Sea spray icing and spray crystallization during powder production of a range of materials requires deep understanding of solidification process occurring in small droplets. To this aim a number of NMR methods have been applied to non-invasive study of freezing process of the droplet as well as to the study of solute redistribution in the final frozen droplet (Thowig et al., 2012). Hindmarsh et al. managed to perform MRI of a 2 mm wide sucrose solution droplet, suspended in cold air that allowed quantifying crystal growth rates and the unfrozen liquid mass fraction in the freezing droplet (Hindmarsh et al., 2004). However, there is no MRI data in the literature concerning the effect of hydrophilicity of substrate on freezing/thawing processes in water droplets. We performed own MRI experiments with water droplet placed on hydrophilic and hydrophobic substrates during freezing/thawing cycling at different temperatures of the processes. It was found out that the surface type affects both the speed of freezing and form of freezing front – in case of hydrophilic substrate the area of water droplet contact with the surface is bigger than those in case of hydrophobic substrate that increases the heat flux (pronouncing the speed of freezing), and slightly curves the freezing front on the thinner edges of the droplet (see Fig. 4). Since the thawing proceeds much slower compared to the freezing, there is practically no difference in thawing dynamics and form of melting front in water droplet placed on hydrophobic and hydrophilic surfaces.

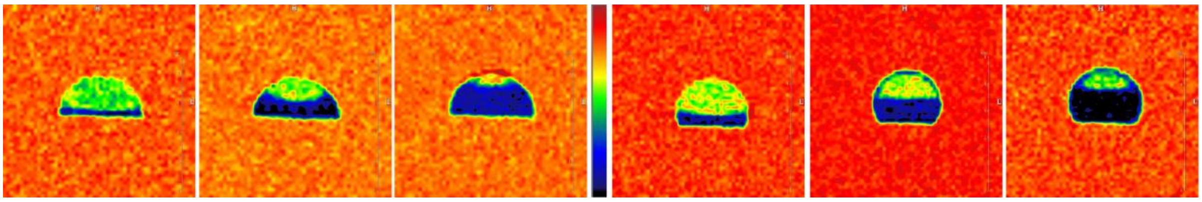

Figure 4. Series of NMR images of a water droplet above the hydrophilic (left row) and hydrophobic (right row) surfaces during freezing at -30°C

As we showed above, MRI appeared very informative for visualization of structural features of normal and salt-water ice, and its ability to quantify the freezing/thawing processes opens up new possibilities for composite materials characterization. Surprisingly, MRI of ice in form of composite structure was considered only in a few articles, which considered some MRI experiments. We could not find any review describing the systematic applications of this method to studying reinforced ice and ice-based composites. Thus, the majority of experimental works are focused on MRI monitoring of freezing/thawing processes occurring in food and plants (Kerr et al., 1998; Ide et al., 1998). For example, MRI was applied to real-time study of the freezing of raw potato and it was found that there is no sharply defined ice front moving through the tissue, but rather a region where various fractions of ice and liquid water co-exist and extent progressively (Hills et al., 1997). The only work where the ice formation in structural composite materials was observed via MRI is a paper of Balcom et al., where they studied freezing front propagation into concrete structures (Balcom et al., 2003).

We decided to fill the gap and perform a systematic MRI study of freezing/thawing processes in different materials where ice can be viewed as a matrix of composite structure. First of all, we studied the model ice systems such as glass beads – ice, glass fiber – ice, and sawdust – ice (Pykrete) composites. Despite quite different nature of reinforcing material, a little difference in qualitative character of freezing/thawing processes was actually found. In every case the formation of ice during freezing or formation of liquid water during thawing follows the common pattern – beginning from the outer areas of the sample the freezing/melting front propagates forward until the process is completed (see Fig. 5). Yet the temperature of the thawing determines clear visualization of the frozen part of the composites; for example, if the temperature is relatively low, the melting front propagates slowly and ice in the inner part begins to thaw ahead of melting front, giving rise to an enhanced NMR signal in this part.

Due to clear visualization of melting/freezing fronts during the processes, it becomes possible to quantify the rates similarly to those described above for salt-water ice. The only difference here is the parameter for quantification – clear boundaries provide accurate measurements of area, *S*, of frozen/thawed part inside the composite samples and its evolution in time (see Fig. 6). In some cases it is possible to measure the linear dimensions of front propagation, i.e. time dependence of length passed by the front. Recently, we showed reasonable agreement between the results of experimental MRI measurements and those predicted by calculation (Stefanovskiy, et al., 2019) that proves the correctness of quantitative data.

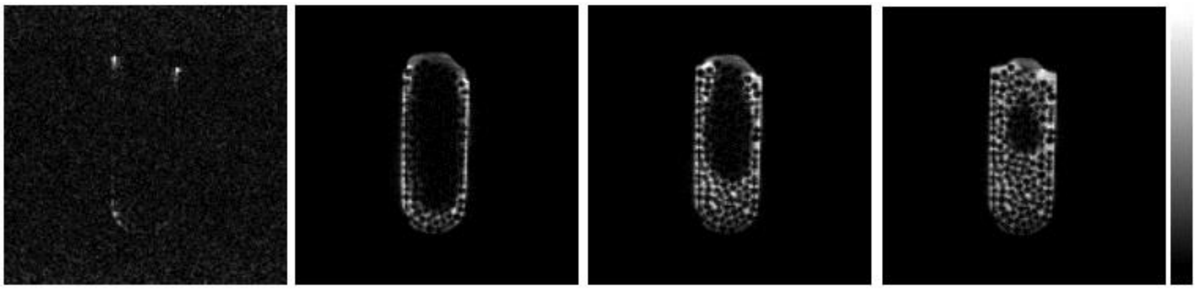

Figure 5. Series of NMR images of glass beds – ice composite during the thawing process at temperature +20°C

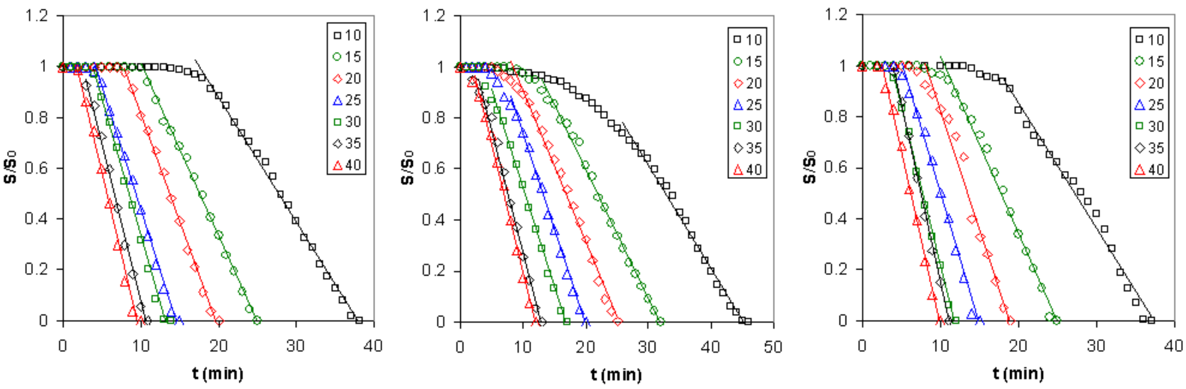

Figure 6. Time evolution of relative area of frozen part for ice reinforced by (left to right) sawdust, glass fiber, and glass beads at different temperatures of thawing

Presented approach was extended to different materials, which form the ice-based composites to provide materials characterization and to reveal the factors affecting the thermal behavior of reinforced ice. Thus, MRI study of freezing/thawing of highly porous ceramic materials filled by water showed strong effect of porous matrix density on thawing/freezing front dynamics whereas filling of materials macropores by aerogel does not affect the dynamics. Similar MRI study of nonwoven polymer fibrous materials filled by water demonstrated interesting effect – introduction of the hydrophobic fiber into composite structure makes the freezing/thawing front propagation slow down due to discontinuity of heat-conducting media.

The methods of ice reinforcement by macrostructural network (either disperse or fibrous) are now actively complemented by introduction of the nano-sized fillers such as colloids and hydrogels. These nano-sized reinforcing agents affect the microstructure of ice resulting in superior mechanical strength and low filtration factor (Vasiliev, et al., 2012; Gunko, et al., 2013). Consequently, MRI study of freezing/thawing processes in ice reinforced by nano-sized fillers is of particular interest. MRI study of freezing/thawing of water suspensions of alumina nanofibers clearly revealed the effect of nanofibers concentration on the morphology of reinforced ice. Thus, the formation of ice in 10 wt.% suspension becomes more homogeneous, with a high degree of continuity; during the melting front propagation the internal regions of thawing or disintegrity of the entire structure are not visualized(see Fig. 7). However, despite the obvious structural effect, the quantitative dynamics of the freezing/thawing in suspensions of nanofibers remain almost unchanged.

Slightly different picture was observed in frozen hydrogels. MRI studies of the freezing/thawing processes in polyacrylic acid hydrogels showed that the introduction of macromolecular reinforcement leads to a significant decrease in the rate of freezing/thawing front propagation in comparison with pure water (ice) and suspensions of alumina nanofibers. At the same time, the mass content of swollen hydrogel in the system has a little effect on the rates due to insufficient change of thermophysical characteristics of the material. Using the charged macromolecular network (sodium polyacrylate instead of polyacrylic acid) changes the morphology of forming ice, (see Fig. 7) – thawed ice demonstrates strongly inhomogeneous structure likely caused by formation of local brine inclusions and subsequent depression of melting point.

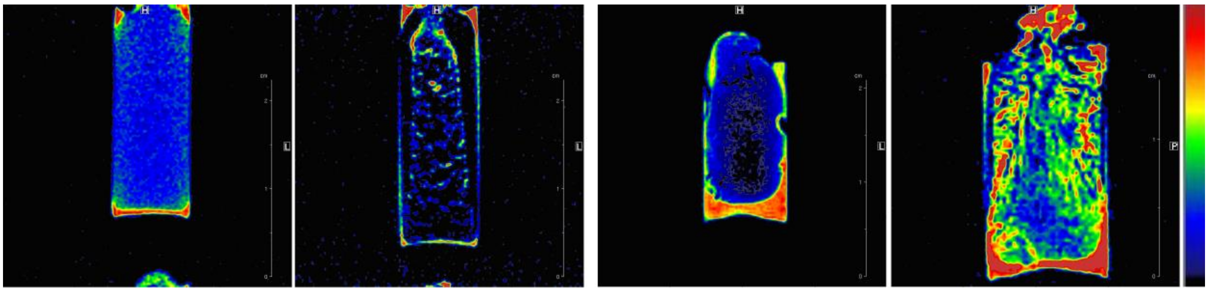

Figure 7. NMR images showing the difference between ice morphology in frozen suspension of Al_2O_3 nanofibers with 10 wt.% and 1 wt.% (left couple), and frozen hydrogels of polyacrylic acid and sodium polyacrylate (right couple)

CONCLUSION

The paper presents numerous results of MRI study of ice and its composites with the aim to systematize different study areas associated with problems of ice, freezing, and thawing in Arctic region. Brief literature review was included to introduce the main achievements of MRI in this field, advantages and limitations of the method in respect to ice research, and new problems and challenges for the ice science. Further we presented the results of own experiments, which briefly describe the main applications of MRI to normal ice, salt-water ice, reinforced ice, and ice-based composites. The results obtained demonstrate the advantages of MRI method in the study of the ice composite materials promising for the Arctic region. In addition, new data on the structure and properties of ice composite materials effectively complement the results of classical mechanical tests of such composites for the purpose of their comprehensive and complex study for further practical applications.

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