Factors affecting quenching in cryogenic liquids

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Abstract. The present paper considers new experimental results on quenching of stainless steel spheres in liquid argon and nitrogen. The experiments were performed on polished, hoarfrost and iced surfaces. The experiments on hoarfrost surface demonstrated higher cooling rates in comparison with corresponding experiments on polished and iced surfaces. This result could be explained by the model of incipience of highly intensive film boiling regime in subcooled liquid.

1. Introduction
Cooling of bodies in cryogenic liquids is a widespread technological process, which finds application in many branches of industry. For example, in the food industry, the use of accelerated shock freezing increases the production efficiency. But the most part of cooling as a rule conducted in stable film boiling regime with low intensity. Thus, the most of investigations are aimed at revelation of possible approaches to intensification of film boiling regime.

When we deal with a cryogenic liquid, it is very difficult to perform quenching at subcooled conditions because there is a very narrow range of temperatures, in which nitrogen or argon stays in a liquid state. So, the real way to impact the cooling process is to change the properties of heat transfer surface.

In paper [1], experimental results on quenching of highly superheated copper plate with low thermal conductive coatings (vacuum Ramsay grease) in liquid nitrogen were presented. The coating thickness was varied from 0.09 up to 0.67 mm. The main result is that low thermal conductive coatings have a significant effect on the character of the temperature curves and total time of plate cooling. Total cooling time for the copper plate with coating was three times lower than for the bare copper plate. The main reason of cooling acceleration is high surface temperature, at which stable film boiling changes into high intensive boiling mode. The transition temperature was higher than the critical temperature of nitrogen. The authors rightly noted that the influence of wettability should not be important in this process due to high wettability of liquid nitrogen both for copper and coating. Later, this work resulted in a series of research works devoted to rewetting of a vertical copper heater with a low-thermal-conductive coating by a falling nitrogen film [2, 3]. It was shown that the coating decreases the total cooling time of plate more than fourfold. These results show the connection of physical nature of increasing transition temperature at intensive boiling regime incipience between pool quenching and falling film cooling. The only condition is the nonstationarity of the process.

Recently, the authors from China [4] performed experiments on cryogenic quenching of rocks using liquid nitrogen. It was shown that the pores, cavities and low thermal conductivity coating may increase temperature corresponding to the beginning of the intensive boiling mode compared to bare copper surface.

The process of metal balls quenching in liquid nitrogen was investigated by Mazor with co-workers [5]. They created a frost layer on the surface of a cooled copper sample from ambient moist air. The authors measured a thickness of frost layer as function of the initial surface temperature, different air velocities and air humidity. The highest thickness of the frost layer did not exceed 200 μm. The main
result was in a great increase of cooling process for samples with frost coating. The efficiency of heat transfer grows as a function of the frost layer thickness. Experiments, presented in the current paper differ from [5, 6] by using argon as a cooling liquid, stainless steel as a sample material and iced formation as a coating.

We should note that all papers discussed above do not consist any physical models which can explain such great increase in transition surface temperature. The current paper is aimed to interpret the results of high intensive cooling of samples with low conductivity layer in cryogenic liquids by using the approximate model of incipience of highly intensive film boiling regime in subcooled liquid [7].

2. Experimental facility

Experiments were performed on a special facility developed for studying the quenching process in cryogenic liquids. The facility is schematically depicted in Fig. 1.

![Schematic of the experimental facility](image1)

The special vessel for cryogenic liquid is a container with a double bottom. The space between the walls of the vessel is filled with the same cryogenic liquid as in the inner volume (1). Using “water bath” principle helps to avoid boiling in the main vessel. The tested sample – metal ball (2) is heated in a coil (3) of HF inductor (4) and immersed into the vessel (1) filled with cooling liquid (liquid argon or nitrogen). The temperature is measured by type-K thermocouples embedded in the tested sample. During cooling, the signal from the thermocouples (6) passes through the connector NI SCXI-1303 on measuring module NI SCXI-1102 (7), which is a part of the assembly based on the NI SCXI-1001. Signal detection from each thermocouple is carried out at frequency of 100 Hz. The measurement results are transmitted through USB interface to the personal computer 8, where temperature dependence on time is plotted in LabVIEW.

![Photo of surfaces](image2)

Fig. 2. Photo of surfaces, used in experiments: a-polished, b-hoarfrost, c-iced
A stainless steel (AISI 316) sphere of 40 mm in diameter was used as a tested sample. The sphere is equipped with five cable thermocouples (one in the center and four on the surface). The experimental facility and technology of test pieces manufacturing are described in detail in our previous work [9]. In experiments, were used three types of surfaces: polished, hoarfrost and iced. The typical height of asperities on polished surface was 5 μm (Fig. 2a). Hoarfrost surface (Fig. 2b) was created by exposing the preliminary cooled ball in the ambient humid air. The exposition time was 30-60 s. The maximal height of iced asperities was less than 1 mm. Iced surface (Fig. 2c) was created by rapid immersing of preliminary cooled ball (-100 °C) into water (20 °C). To prevent weak adhesion of ice with a smooth steel surface, first, it was treated with a spray of water from an atomizer to create asperities. The typical thickness of iced layer was 1 mm. The surface was rather smooth and robust.

3. Results and discussion
As it was mentioned above, liquid argon (LA) and liquid nitrogen (LN) were chosen as a coolant liquids. The experiments on polished and hoarfrost surfaces were performed in LA, and experiments on polished, hoarfrost and iced surfaces were performed in LN.

Experimental thermograms obtained during cooling of steel ball in LA are presented in Fig. 3. These thermograms are the averaged surface temperatures (4 point on surface) depending on time.

![Fig. 3. Typical thermograms for quenching in LA. 1-polished and 2-hoarfrost surface](image_url)

The initial ball temperature is approximately -20 °C. This temperature is higher by 166 K than the saturation temperature of argon ($T_{s} = -186 °C$) and 100 K higher than the critical one ($T_{cr} = -122 °C$). Such a surface temperature allows us to obtain a region of film boiling regime. As it can be seen in Fig. 3, the cooling process for polished stainless steel ball (1) lasts approximately 300 s. A large part of the process passes in stable film boiling regime and lasts 250 s, the typical cooling rates is about 0.5 K/s. The results obtained on the same ball with hoarfrost layer are very different from a polished ball. The intensive heat transfer regime is observed immediately after sample immersion. The stable film boiling regime is absent, so, the cooling rates are very high and reach 6.4 K/s. Total cooling time is about 30 s that is 10 times less in comparison with the results obtained with the polished surface.

Based on thermograms, which were used in solving the inverse heat conduction problem, we calculated a heat transfer coefficient (HTC) on a ball surface. These results are presented in Fig. 4. One can notice that the values of HTC for polished sample are in a good agreement with equation for stable film boiling regime of subcooled liquids, obtained by authors of [10]:

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This equation allows defining HTC for film boiling of liquids at any temperatures. For our case we used \( \Delta T_{\text{sub}} = 0.1 \) to satisfy the condition of saturated liquid. The values of HTC obtained by Eq. (1) do not exceed 200 W/(m\(^2\)K). But the values of HTC for the sample with hoarfrost layer are much higher: the typical values are about 1000 W/(m\(^2\)K) (Fig. 4 (2)). It means that on the ball with hoarfrost we have observed a particular boiling regime, which differs from stable film boiling regime by rates of HTC by an order of magnitude.

\[
\alpha = 0.59 \left( \frac{\Delta T_{\text{sub}}}{\nu \Delta T_{\text{h}}} \right)^{1/4} \left( 1 + 43.5 \left( \frac{\mu_{\text{g}} h_{\text{TG}} g \Delta T_{\text{sub}}}{\lambda_{\text{g}} \Delta T_{\text{h}} \cdot \operatorname{Pr}_{\text{g}}} \right)^{1/2} \frac{\Delta T_{\text{sub}}}{\Delta T_{\text{h}}} \right)^{1/2} \left( \frac{\rho_{\text{g}}}{\Delta T_{\text{h}}} \right)^{1/8}
\]

(1)

\( \Delta T_{\text{sub}} = 0.1 \) to satisfy the condition of saturated liquid.
The initial temperature of the samples was -30 °C. We have observed stable film boiling regime, which lasted approximately 250 s, for the sample with polished surface. Cooling rates in this regime are very low. Experiments on hoarfrost surface are similar to those on LA. The cooling process becomes very intensive that is not typical for quenching in cryogenic liquids. The surface temperature falls by 160 K in only 25 s. But experiments on stainless steel ball with iced surface (Fig. 5 (3)) are quite similar to the polished one. Cooling lasts 260 s with the rates similar to the polished surface. The iced coating differs from the hoarfrost by relief (porous and smooth), density and heat conduction coefficient. This result shows that the structure and thermophysical properties have a great influence on a cooling process.

Fig. 6 presents HTC obtained from thermograms depicted in Fig. 5. HTC for polished and iced surfaces are similar and not higher than 200 W/(m²K). But HTC for hoarfrost surface is by an order of magnitude higher and the typical values are 1000 W/(m²K). All the results are similar to those, obtained on LA.

![Graph showing HTC comparison (1 - polished, 2 - hoarfrost, 3 - iced)](image)

The model demonstrates an effect of thermal effusivity of a cooled metal sphere and explains the results, obtained with the hoarfrost surface: the lower thermal effusivity of surface and the higher temperature when high intensive boiling regime arises.

The main idea of the model is that the liquid wave can periodically touch surface roughness protrusions; if their temperature appears to be lower than the attainable limiting temperature of liquid (T_{lim}), the possibility of direct local contact between liquid and solid wall occurs. Such a possibility can arise due to local cooling of the surface roughness elements, which may differ in chemical composition from the basic metal. These local contacts provide development of the intensive heat transfer regime at film boiling of subcooled liquids. The main calculations are given in the article [8], but the final equation is:

\[
K_o = 4.5 \cdot 10^{-4} \cdot \frac{h_{LG} \sigma}{g \rho} \left( \frac{D}{v} \right)^{1/2} \left( \frac{\rho \sigma}{D^2} \right)^{7/16} \left( \frac{Pr^{7/16}}{Gr^{7/16}} \right) \left( \frac{\Delta T_{sub}}{\Delta T_w} \right)^{1/2}.
\]

The model demonstrates an effect of thermal effusivity of a cooled metal sphere and explains the results, obtained with the hoarfrost surface: the lower thermal effusivity of surface and the higher temperature when high intensive boiling regime arises.
Fig. 7 shows the relation between complex \((\rho c_\lambda)\) of the cooled surface and temperature corresponding to the high intensive boiling regime beginning. It is shown that for \((\rho c_\lambda) \sim 10^4\), this regime occurs in the temperature range similar to those observed in experiments (from -50 to 0 °C). It is possible to find the values of thermophysical properties of hoarfrost: \(\rho \approx 100 \text{ kg/m}^3\), \(c \approx 2000 \text{ J/(kgK)}\) and \(\lambda \approx 0.1 \text{ W/(mK)}\). According to the given properties, the complex \((\rho c_\lambda)\) is \(2 \times 10^4\) approximately, which confirms our estimates. Of course, it is only estimates, and the strict determination of surface properties is in a plan of our future investigations.

These researches could help to introduce innovative improvements in the food industry at the phase of shock freezing of products. Acceleration of cooling process will increase productivity of equipment, which could save production space and man-hours. This could directly affect the final cost of the product. Such a competitive advantage allows manufacturers to win the price struggle and increase in food quality standards.

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