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Evaporation heat transfer of liquid nitrogen on microstructured surface at high superheat level

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ABSTRACT

Liquid nitrogen, as a coolant, is generally applied in cell vitrification cryopreservation. It takes heat from the carrier with cell samples through its violent evaporation on the carrier surface. As a result, the temperature of the carrier plunges dramatically. This article focuses on the unsteady evaporation heat transfer characteristics of liquid nitrogen on a microstructured surface etched into the frozen carrier surface at a high superheat level. The heat flux and evaporation heat transfer coefficient of liquid nitrogen were investigated using a lumped capacitance method. The experimental results showed that the cooling rate of the thin film evaporation on the microstructured surface is obviously higher than that of pool boiling, which is currently being used for cell cryopreservation. The heat flux and the evaporation heat transfer coefficient work together to present a parabolic trend with the superheat decreasing during this heat transfer process. Besides, the microstructure of the surface has an important effect on the evaporation heat transfer of liquid nitrogen. The larger the thin film evaporation zone is, the higher the heat transfer coefficient is. The current investigation results in a cell cryopreservation method through vitrification with relatively low concentrations of cryoprotectants.

1. Introduction

Liquid nitrogen, as a coolant, is generally applied in cell vitrification cryopreservation. It takes heat from the carrier with cell samples through its violent evaporation on the carrier surface. As a result, the temperature of the carrier plunges dramatically to − 196 °C, the temperature of liquid nitrogen. In this process, a freezing rate is a key parameter. A faster freezing rate can effectively improve the cell survival rate. With improvements, it has increased to 20,000 °C/min when using open pulled straws (OPS) [1,2], to 24,000 °C/min using an electron microscopic (EM) grip [3,4], and to 20,000 °C/min using a cryoloop [5]. Although these methods achieve complete or partial vitrification of cell samples, highly concentrated cryoprotectants are required. Improving the freezing rate can further decrease the concentration of cryoprotectants and thereby increase the cell survival rates.

From the perspective of heat transfer, the main obstruction to further increasing the freezing rate is that liquid nitrogen vaporizes near the carrier surface during pool boiling while directly plunging carriers into liquid nitrogen such as the EM grip, OPS, and cryoloop methods. Film boiling takes place because of the huge temperature difference between the carrier surface and the liquid nitrogen. The evaporation forms a "vapor blanket," which acts as a heat-insulation layer as shown in Fig. 1. As a result, the heat transfer coefficient between the sample surface and liquid nitrogen is limited (< 1000 W/m^2 °C [6]) due to the poor thermal conduction of the "vapor blanket." Therefore, the removal of "vapor blanket" is critical for further improving the freezing rate when vitrification is used for cell cryopreservation. Steponkus et al. [7] and Yoon et al. [8] attempted to further cool liquid nitrogen and transformed it into a slush state at a lower internal temperature of − 210 °C by applying a negative gauge pressure. In their experiments, *Drosophila melanogaster* embryos and human oocytes were plunged directly into the slush nitrogen and cooled by the solid-liquid phase change instead of the liquid-vapor phase change. Both groups came to the same conclusion that the survival rate of the sample can be improved. However, neither of them measured the freezing rate using such methods. Su et al. [9] designed a carrier with a microstructured surface and developed an ultra-fast cooling method to utilize thin film evaporation instead of pool boiling to remove the vapor blanket. The freezing rate utilizing thin film evaporation reached 49,045 °C/min. Thin film evaporation can significantly increase the evaporating heat transfer coefficient [10–14]. The heat transfer coefficient can reach an order of magnitude of 10^6 W/m^2 K in the thin film region of the microstructured surface [15].The temperature difference between the

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Nomenclature

Bi	Biot number
C_p	specific heat capacity of silicon, J/kg °C
d_1	thickness of silicon base, m
d_2	thickness of microstructured layer, m
h	evaporation heat transfer coefficient, W/m^2 °C
k_1	conductivity of silicon, W/m °C
k_2	conductivity of microstructured layer, W/m °C
k_N	conductivity of liquid nitrogen, W/m °C
m	mass of frozen carrier, kg
Q	heat quantity, J
q	heat flux, W
R_{cond}	conductive resistant, m^2 °C/W
R_e	evaporation heat resistant, m^2 °C/W
R_t	total heat resistance, m^2 °C/W
R_1	conductive resistance of silicon base, m^2 °C/W
R_2	conductive resistance of micro structured layer, m^2 °C/W
R_3	evaporation heat resistance of liquid nitrogen, m^2 °C/W
s	evaporation heat transfer area
T	Temperature, °C
t	time, s
ε	proportion of liquid nitrogen in microstructured layer

carrier and the liquid nitrogen is very large. It is > 200 °C at the beginning of the freezing process. Besides, the temperature difference decreases at a rapid pace from 200 °C to 0 °C within a very short time that is < 0.4 s; that is, the process is an unsteady heat transfer process. In this investigation, the evaporating heat transfer process has been studied with a focus on the unsteady evaporation heat transfer of liquid nitrogen onto a microstructured surface including the effect of the surface microstructure of the carrier on the instant evaporation heat coefficient.

2. Experimental system and procedures

2.1. Experimental system

An experimental system was designed and is illustrated in Fig. 2. This system includes a 1.5 m^3 vacuum chamber with a vacuum pump, a frozen carrier, two CRY-AC B-700 liquid nitrogen containers (Brymill Cryogenic Systems, Ellington, CT, USA) connected with liquid nitrogen jet tubes (2 mm inner diameter, 291 mm length), an Absolute Piezo Transducer Series 910 pressure sensor (MKS Instruments, Inc., Longmont, CO, USA), a fine T type thermocouple of 70 μm in diameter (OMEGA Engineering, Inc.), and a data acquisition system (National Instruments).

The vacuum chambers and pumps provided the vacuum environment for the experiment. The vacuum chamber volume was large enough to provide a pressure buffer so that the pressure would not increase dramatically when the liquid nitrogen evaporated. The liquid nitrogen containers injected the liquid nitrogen into the vacuum chamber and sprayed it onto the surface of the frozen carrier. During the cooling process, the pressure sensor and thermocouples would measure the actual pressure and temperature and send the information to the data acquisition system. The data acquisition rate was 1000 n/s. The uncertainty of the measured temperatures is ± 0.3 °C. The uncertainty of the measured pressure is ± 50 Pa.

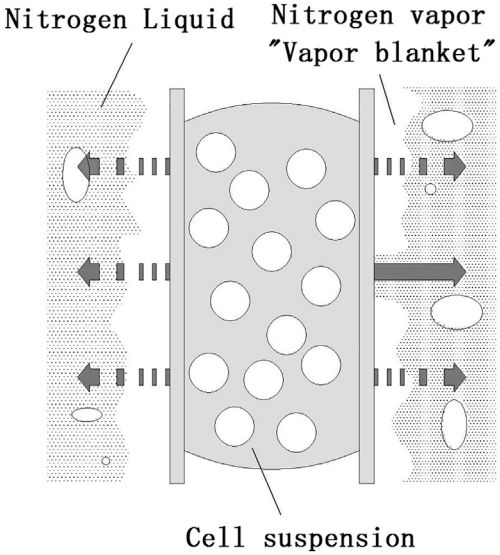

Fig. 1. Schematic of the formation of the "vapor blanket" during the pool boiling of the OPS method [9].

The frozen carrier is used to hold the cell sample and to increase the freezing rate via thin film evaporation. As shown in Fig. 3, the carrier was made from two silicon chips. Each chip has a length and width of 10 mm and a thickness of 300 μm. A 100 μm thick gap lies between the two silicon plates which is used to hold the cell sample. A thermocouple is placed in the centre of the gap. The microstructured surface consists of a cylindrical array, which is etched into the other side of the chip. The cylindrical array allows enough capillary force to support better surface wetting and to gain a larger area of liquid thin film. To optimize the evaporation heat transfer, four micro structured surfaces were produced. Their structure parameters are shown in Table 1. Square and circular pillars are embedded in the surfaces respectively. Their micrographs are shown in Fig. 4. The square pillar has a cross-section of 100 μm × 100 μm with a height of 100 μm. The circular pillar has a diameter of 100 μm with a height of 100 μm.

2.2. Experimental procedure

First, the frozen carrier and the thermocouple were put in place, along with the liquid nitrogen spraying devices, the pressure sensor and the data acquisition system. Then a vacuum was pulled onto the vacuum chamber. The absolute pressure was 10.30 kPa. In the experiments, this pressure increased slightly to 10.65 kPa because of the limited volume of the vacuum chamber and the evaporation of liquid nitrogen. The two valves on the liquid nitrogen containers were opened simultaneously, directing the liquid nitrogen flow to the frozen carrier.

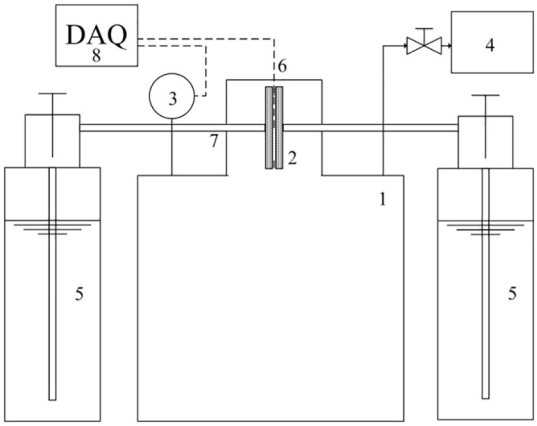

Fig. 2. Schematic of experimental system (1. vacuum chamber, 2. frozen carrier, 3. pressure sensor, 4. vacuum pump, 5. liquid nitrogen container, 6. thermocouples, 7. liquid nitrogen jet tubes, and 8. data acquisition system).

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