Experimental study on heat transfer characteristics of liquid nitrogen through coated transfer line

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Abstract. The effective and efficient utilisation of cryogen is made possible by optimised transfer lines which ensures reduction in chilldown time and cryogen usage. Cryogenic chilldown experiments were conducted on coated and regular copper tubes. Liquid nitrogen at different mass flux condition was used as the working fluid and Polyurethane coatings belonging to the family of Teflon was used as coating material. The performance of the regular and coated copper tubes at corresponding mass flux were compared. An increase in heat flux and heat transfer coefficient along with reduction in chilldown time was obtained by providing a low thermal conductive coating between the cryogen and wall material.

1. Introduction
Cryogen are widely being used in many industries for the purpose of food processing, material recycling, biological preservation etc. The cryogens will be stored in Dewar vessels and transported to the point of usage through transfer lines made of different materials. The process of Cryogen transport is so complicated since it incorporates intricate heat transfer between the wall material and cryogen, resulting in two phase flow and pressure surges. The identification of flow and heat transfer characteristics is badly at need in those industries which make of cryogens. Since the process involve two phase flow, the detailed heat transfer occurred during transport of cryogens are not well understood. Under chilldown conditions, a steady state will be attained between the transfer line walls and the cryogen being transported. A broad research on the flow structure and heat transfer characteristics are required to develop widespread understanding of this complicated phenomena. Earlier research works suggest reduction in time taken for chill down process during the cryogen transport across coated transfer lines[1]. Present study deals with heat transfer characteristics in coated transfer lines made of copper, coated with a low thermal conductive coating belonging to the family of Teflon.

2. Literature Review
According to Cowley et al.[2], reduction in chilldown time can be attained by incorporating a low thermal conductive coating between the cryogen and pipe wall material. According to Allen et al.[3]response of cryogenic systems can be improved by providing such internal coatings on cryo-panels which in turn reduce the time taken for chilldown. Reed et al.[4] made a comparison between regular and internally coated propellant lines and identified faster cool down in coated lines.
In room temperature liquids, the modification in transition boiling could be attained by interposing thin insulating layers on transfer line wall [5]. Gaertner [6] witnessed reticence of Leidenfrost phenomenon in coated cryogenic transfer lines. According to Leonard et al. [7] coated line chilldown is independent of cryogen flow rate, where shift to the nucleate boiling region is attained earlier. After a short period of time steady state will be attained there by indicating rapid cool down. Driester [8] conducted heat transfer enhancement using low thermal conductive materials which allows deep filling of the conduits with cryogens. This effectively shorten the time taken for chilldown to three times and will also significantly diminish the heat transfer surface of cryogenic liquid evaporators. The use of low conductive coatings permits to obtain the FB crisis at considerable temperature drops and its transition with more effective heat transfer. The use of low-conductive coatings on the internal tube surface resulted in thrice reduction of tube precooeling time.

Hong Hu et al. [9] performed an experiment to evaluate the modification and enhancement on the quenching heat transfer by a nano porous heat transfer surface. The results indicated that the nano porous surface completely modified and enhanced the phase-change heat transfer in all three quenching regimes. Enhancements are mainly attributed to the super hydrophilic property and nano scale nucleation sites offered by the nonporous surface. Kim et al.[10] Studied the effect of surface roughness on pool boiling heat transfer coefficient and critical heat flux of copper surface having moderate wettability. It was confirmed that surface roughness enhances nucleate boiling heat transfer because of the increased number of active nucleation sites. CHF exhibited considerable augmentation with increasing surface roughness. In the same year Li-Wu et al. [11] conducted an experiment on super hydrophilic surface realized by enhancing the surface roughness with silica nano-particles on SS spheres following the spray coating method. The application of such surface to quenching in water was tested under both saturated and sub cooled conditions. It was shown that quenching is accelerated on the super hydrophilic surface by a factor of 60%, due to the marked boiling heat transfer enhancement with the critical heat flux being increased by 78%.

In 2017, Daisu et al. [12] experimentally investigated a method for reducing the time and total mass of cryogenic fluid required for a chill down process for piping with Poly tetra fluoro ethylene coating. The results indicated that the temperature of the minimum heat flux point was higher for the pipe with insulating layer. This resulted in the decrease in chill down time and total mass of LN2 consumed in the chill down process. In the same year Ran Li and Zhongwei Huang [13] developed a new CHF model for saturated pool boiling on surfaces with micro-scale roughness, including micro-pillar, micro-ridge structures as well as random roughness made up emery paper or sand papers. The model accounted for the effects of roughness-augmented wettability and capillary wicking on CHF enhancement. In 2017, Shoji Moria and Yoshio Utaka [14] proposed numerous surface modifications to enhance the critical heat flux (CHF) in a saturated pool boiling. CHF enhancement is obtained as a result of effects of extended surface area, nucleation site density, wettability, capillary wicking, and wavelength decrease based on the modified Zuber hydrodynamic stability model.

Therefore, if the existence of a liquid meta-stability limit is presumed, a minimum in cool down time may be predicted to occur at an optimum coating thickness and is in qualitative agreement with observed behavior. These observations showed that it is essential to lower the coating surface temperature close to the meta-stability limit and to know more about time-averaged values of the vapor heat transfer coefficient. Therefore, further investigations have been undertaken with a technologically feasible coating material. Considering the theoretical aspects of the vapor film formation process, subsequently with the test specimens and experimental procedures, results and discussion has been made and arrived at conclusions concerning the unknown thermo hydrodynamic details of the cool down process.

3. Experiment work

Figure 1 shows the schematic of experimental set up which consists of Liquid Nitrogen Dewar vessel, Gaseous Nitrogen cylinder pressure regulator, valves, DAQ system, electric heater and flow meter. The test sections used for the experiment, was a normal copper tube [internal diameter 5/16 inches (0.0079m) OFHC Copper tubes (UNS C10100)] and second the copper tube with its interior coated with a thermally
low conductive polyurethane coating. The pipe was coated with a simple method of fill-and-drain, creating a very thin coating. The tube was then placed vertically overnight to dry and solidify the coat. Thermocouples were attached at lengths 0.02 m, 0.07 m, 0.12 m, and 0.17 m from the inlet side. At each point three thermocouples are attached at 120° apart. The heat in-leak to the test section was minimized using a thick layer of polyurethane foam insulation (thermal conductivity of 0.02 W/m K, density of 11 kg/m³) which is a type of expanded foam insulation. Test section was also covered by yarn and then by Nitrile rubber insulation.

Liquid Nitrogen was supplied to the test section through 1/2” SS 304 grade pipes and brass fittings from a 55 L Dewar made by IBP Co. Limited (TA-55). The liquid nitrogen was pumped from Dewar vessel by external pressurization using a gaseous nitrogen cylinder and having 47 L capacity. Flow rate of liquid Nitrogen was manually regulated by a pressure regulator mounted on external pressurization line. Initially the entire test section was purged with gaseous nitrogen. A by-pass line is introduced to ensure the entry of saturated LN2 into the test section. Temperature measurement of the test section was

![Diagram](image)

**Figure 1.** (a) Schematic of Experiment Set up. (b) Test section.
done using T-type thermocouples connected to Keysight 34972A data acquisition / data logger switch unit with scan frequency of 30 milliseconds which was also connected to the personal computer for data analysis. The average mass flux was measured using a Single phase volume flow meter having an accuracy of 0.05 mm3/s at the exit line from the test section. To ensure single phase gas flow entering the flow meter, the outlet line is placed in hot water bath heated by electric heaters. Pressure gauge is provided for measuring and regulating initial pressure. Different flow control valves are provided to control the flow.

4. Results and discussion

4.1 Time- Temperature comparison for different mass flow rates

![Figure 2. Temperature profile at different inlet pressures for coated and regular copper tube.](image-url)

Table 1. Percentage saving in film boiling time For copper tube.

| Mass Flux (kg/m²s) | Test Section     | Transition Temperature (K) | Transition Time (s) | % saving |
|-------------------|------------------|----------------------------|---------------------|----------|
| 66 kg/ m²s        | Coated copper Tube | 180                        | 227                 | 13.2 %   |
|                   | Regular copper Tube | 154                        | 257                 |          |
| 86 kg/ m²s        | Coated copper Tube | 184                        | 125                 | 7.2 %    |
|                   | Regular copper Tube | 165                        | 134                 |          |
| 102 kg/m²s        | Coated copper Tube | 183                        | 105                 | 2.8 %    |
|                   | Regular copper Tube | 160                        | 108                 |          |

At the mass flow rate of 66 kg/ m²s, coated transfer line requires 225 seconds to cover the transition boiling regime, the temperature corresponds to this time is 170 K. The corresponding values for regular tubes were 253 sec and 158 K. At the mass flow rate of 86 kg/ m²s, coated transfer line requires 119 seconds to cover the transition boiling regime, the temperature corresponds to this time is 183 K. The
corresponding values for regular tubes were 130 sec and 157 K. At the mass flow rate of 102 kg/m²s, coated transfer line requires 99 seconds to cover the transition boiling regime, the temperature corresponds to this time is 188 K. The corresponding values for regular tubes were 101 sec and 161 K. It can be seen that, with increase in mass flux, there is a substantial reduction in the time taken for transition.

For the regular tube, the solid-fluid thermal resistance forms the basis for the heat transfer at the interface. The vapor film developed immediately between the solid wall and fluid during the initial flow period causes an increase in the time required for chilldown as it takes time to break this boiling envelope.

In the case of coated surfaces, the low conductivity of the coating material results in a thermal gradient on its either side causing a reduction in temperature between fluid-fluid interfaces. Thus, resulting in reduction of chilldown time. The results presumed that a considerable reduction in chilldown time can be obtained by providing a coating of low conductivity on the inner walls of the conducting tubes and for any mass flow rate condition, the coated transfer line covers the film boiling regime at a higher wall temperature as compare to the regular tube. This results in a reduction in total chill down time since more than 75% of the total chill down time is covered for clearing the film boiling regime [15]. Table 1 shows the percentage saving in time for covering the film boiling regime.

4.2 Variation of Heat transfer coefficient

Inner wall temperature can be found by equation

$$T_i = T_o + \left( \frac{\rho c}{4a} \left( r_i \right)^2 - 1 - 2 \ln \left( \frac{r_i}{r_o} \right) \right) \frac{d^2 T_o}{dt^2} + \left( \frac{1}{64a^2} \left( r_i^4 - 5r_i^4 \right) + \frac{\rho c}{8a^2} \ln \frac{r_i}{r_o} - \frac{\rho c}{16a^2} \ln \frac{r_i}{r_o} \right) \frac{d^2 T_o}{dt^2} + ... \tag{1}$$

Inner wall surface heat can be calculated with the equation

$$q_i'' = \rho c \left( \frac{r_i^2 - r_o^2}{2r_i} \right) \frac{dT_o}{dt} - \left( \frac{(\rho c)^2}{k} \left( \frac{r_i^2}{16} - \frac{r_i^2}{16} \ln \frac{r_i}{r_o} \right) \right) \frac{d^2 T_o}{dt^2} + \left( \frac{(\rho c)^2}{k} \left( \frac{r_i^2}{384} - \frac{3r_i^2}{128} + \frac{3r_i^2}{128} \right) \right) \frac{d^2 T_o}{dt^2} - ... \tag{2}$$

Heat transfer coefficient can be found using the equation

$$h_i = - \frac{q''}{(T_i - T_{sat})} \tag{3}$$

Table 2. Uncertainty analysis.

| Measured parameters       | Uncertainties |
|---------------------------|---------------|
| Length, L                 | ±1 mm         |
| Wall thickness            | ±0.01 mm      |
| Temperature, K            | ±0.15 K       |
| Pressure                  | ±1.2 kPa      |
| Volume flow rate          | 0.05 mm3/s    |
| Maximum deviation in q''  | 12.8 %        |
| Maximum deviation in h_i  | 10.7 %        |

Burggraf [16] in the year 1964 developed a method for the estimation of inner wall temperature from the measured outer wall temperature by using an inverse heat transfer technique. Table 2 shows the uncertainty analysis. The main source of uncertainty are from the temperature, pressure and volume flow rate measurements. Also the uncertainty in the measured values of heat flux and heat transfer coefficients are listed in table 2.

Figure 3(a) and (b) shows the variation of heat transfer coefficient for the un coated and coated transfer line. For a mass flow rate of 66 kg/m²s, the maximum heat transfer coefficient is obtained earlier for
coated section (87 W/m²K) compared to the uncoated section (27 W/ m²K). For the mass flow rate of 86 kg/ m²s, the maximum heat transfer coefficient is obtained earlier for coated section (70 W/m²K) compared to the uncoated section (59.7 W/ m²K). For the mass flow rate of 102 kg/ m²s, the maximum heat transfer coefficient is obtained earlier for coated section (124 W/m²K) compared to the uncoated section (57 W/ m²K).

Figure 3. Variation of heat transfer coefficient with time for (a) un-coated Copper tube and (b) coated copper tube.

4.3 Variation of Heat flux:
Figure 4(a) and (b) shows the variation of heat flux for the un coated and coated copper tube. For a mass flow rate of 66 kg/ m²s, the maximum heat flux attained is 9122 W/m² for the coated section at the same time the corresponding value for the uncoated section is 2091 W/m². For a mass flow rate of 86 kg/ m²s, the maximum heat flux of 5745 W/m² is obtained for coated copper tube and the corresponding value

Figure 4. Variation of heat flux as a function of time for (a) Un coated copper tube and (b) Coated copper tube.
for uncoated copper tube is $4457 \text{ W/m}^2$. Also it can be inferred that the coated tube attains maximum heat flux earlier than the uncoated tube. For a mass flow rate of $102 \text{ kg/m}^2\text{s}$, the maximum heat flux of $10698 \text{ W/m}^2$ is obtained for coated copper tube. Maximum heat flux of $4817 \text{ W/m}^2$ is obtained for uncoated copper tube. Also it can be inferred that the coated tube attains maximum heat flux earlier than the uncoated tube. Thus it can be concluded that for any mass flow rate condition, the coated tubes results in an earlier attainment of maximum heat flux.

**Conclusion**

The test results justifies that the use of Teflon coating in test section will enhance the heat dissipation characteristics with the reduction in cool down time. The data obtained indicates lower chill down time for various inlet pressures, due to the sudden increase in heat transfer after the transition. The obtained data will help in the optimization and design of transfer lines. This work shows the necessity for studying the effects of various coatings on the heat transfer enhancement and chill down time reduction in future. Future works can also employ different coating materials to cool down transfer lines made of stainless steel and other suitable materials. In depth understanding of this phenomena can be obtained by considering the heat transfer coefficient variation in the three flow regimes during chilldown process.

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**Nomenclature**

$d$ Diameter, m  
$q''$ Heat flux, W/m$^2$  
$r$ Radius, mm  
$T$ Temperature, K  
$t$ Time, s  
$\Delta T$ Wall super heat, K  
$\rho$ Density, kg/m$^3$  
$C$ Specific heat, J/(kgK)  
$h_i$ Heat transfer coefficient, W/m$^2$K  
$\alpha$ Thermal Diffusivity

**Abbreviations**

CHF Critical heat flux  
HTC Heat transfer coefficient  
FB Film boiling  
NB Nucleate boiling  
TB Transition boiling

**Subscripts**

$i$ inner surface of wall  
o outer surface of wall  
sat saturation conditions
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