Onset of Heterogeneous Nucleation of HFE-7100 on a Rapidly Heated Micro-Heater at One Atmosphere

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Abstract

The present study deals with the onset of nucleate boiling (ONB) of a superheated liquid due to rapidly heated Micro-Heater (MH) at one atmosphere. A set of experimental results of ONB in HFE-7100 as a result of different heating rates will presented. There are very few studies and thus very scarce amount of data pertaining to rapid heating of HFE-7100 in the literature. In real liquids, namely liquids that are not specially treated for the removal of possible nucleation sites (such as dissolved gasses, suspended particles etc.), a high level of superheating may be obtained using high heating rates such that the liquid's nucleation temperature is substantially higher than its saturation temperature.
Prediction of superheat levels and nucleation temperatures under these conditions as a function of temperature change rate has not been possible with analytic tools up to recently. The present study aims at broadening the experimental data base, in order to gain more understanding on the ONB phenomenon due to rapid heating under various heating rates.

Keywords: Rapid heating; Micro-heater; Experiment; Heterogeneous nucleation; ONB.

Introduction Bubble nucleation inception is a physical phenomenon that characterizes the onset of a phase transition process in a liquid. Spray cooling is one of the most efficient methods to remove heat from surfaces, such as electronics [1]. Mudawar [2] reports that Spray cooling has been identified as a potential solution that can dissipate 150– 200 W/cm2 while maintaining the chip temperature below 125°C, and found HFE-7100 to be an ideal coolant from both thermal management and safety considerations.
The purpose of the research is to predict the nucleation temperature due to an isobaric process under moderate heating rates for Novec™ HFE-7100 dielectric fluid.
When the time required for superheating the liquid is shorter than the time that is required for bubble shedding from the interface, the nucleation temperature (T_{ONB}) is higher than the equilibrium saturation temperature (T_{sat}). In that case, the liquid is considered a metastable liquid, which is a partially, or kinetically, stable system, as opposed to a thermodynamically stable system [1][3]. A deeper penetration into the metastable zone is possible for fast processes. Knowledge of the rate at which a system evolves towards stable equilibrium is essential to any investigation of metastable liquids, because such systems can only be studied over intervals that are short compared to the characteristic time for the appearance of a new phase [4].
In our previous studies, focused on rapid pool boiling, we identified three regions for the nucleation temperature (fast, intermediate and slow heating rates) depending on the heating rate [5][6]. A thermodynamic model, taking into account kinetic issues, was developed for the fast region for various pure liquids at atmospheric pressure (10^6<dT/dt<10^9 K/s) [7]. The model

requires knowledge of the saturation and spinodal temperatures in order to predict the nucleation temperature as a function of the heating rate. Using original and previously published experimental data for water, two correlations valid in the range 0<dT/dt<10^6 K/s for water at atmospheric pressure, were suggested in the follow-up study [5]. For the slow region, a heat transfer model was developed for water at atmospheric pressure under three different levels of subcooling [8]. Recently, a thermodynamic model is developed that resolves the minimal heating rate required for rapid isobaric nucleation at the spinodal limit for both homogenous and heterogenous nucleation was developed [9].
None of these models were examined for HFE-7000. Thus, the present study aims to examine whether the available correlations, developed for other pure liquids, can be used for predicting ONB conditions for rapid heating processes of HFE-7000 at atmospheric pressure.

Experimental apparatus
A micro-Heater (MH) is used to rapidly heat a thin layer of liquid HFE-7100 by Joule heating. The micro-heater is comprised of an array of 96 platinum heaters. A thin layer of Pt (~200 nm) is deposited on a thin layer of Ti that assists in bonding the platinum to the quartz substrate [10]. In the current experiment, only one heater is required. **Figure 1** presents a typical heater (typical size: 122μm x 234μm) with its power leads.

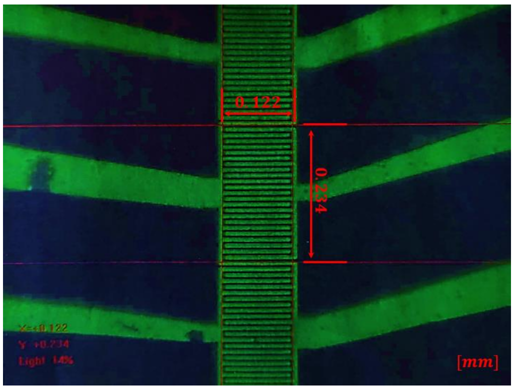

Figure 1. A photograph of a single heater.

Power is supplied to the heater (MH) as is measured instantaneously. The power supply is controlled by either a self-designed pulse-generator or by the high-speed video camera (HSV, Phantom Model), that was connected to the electrical system by a TTL connection. The measurements were stored using a high-speed oscilloscope (PICO 5244D, 200 MHz). The MH is placed in a rectangular Perspex vessel that serves as a container for the liquid HFE-7100. Due to the small size of the heater, and the bubbles at ONB conditions, the high-speed camera is mounted on a microscope (Olympus). The microscope has its own light source and is capable of magnifying up to 50 times (see Figure **2**).

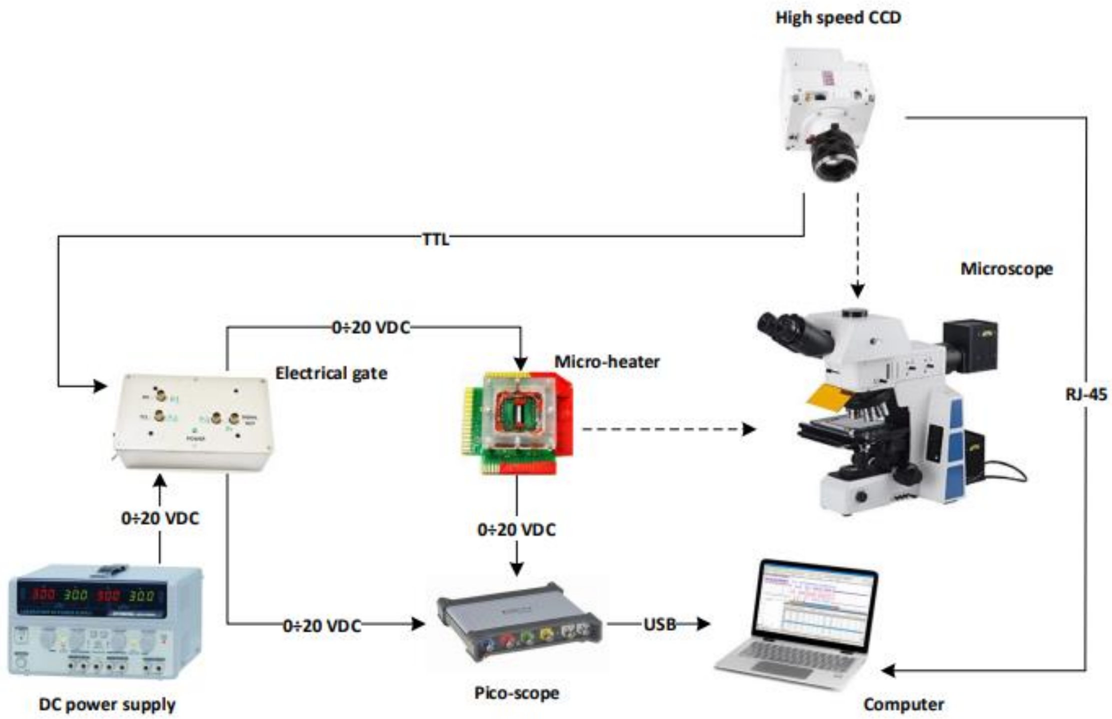

Figure 2. Schematic description of the experimental apparatus.

Calibration

A two-stage calibration procedure was carried out.

I. Thermal characterization of the MH.
II. Characterization of the coupled MH and the electronic system.

I. Thermal characterization of the Micro-Heater (MH).

The micro-heater was placed in a small, controlled oven. The temperature of the oven was increased by 1°C/min, which is slower than the thermal response time of both the MH and of the exposed PT100 sensor that was used to measure the temperature in the vicinity of the MH. A NETDAQ 2645A data logger was used with a 4-wire connection to measure the temperature. A 2-wire method was used to measure the resistivity of the MH. Four heating and natural cooling cycles were conducted to make sure that the MH is properly annealed and that no hysteresis prevails. The electrical resistance of the MH is linear to its temperature, as shown in **Figure 3** and the obtained slope (0.00206 1/°C) is equals to a similar platinum MH [10].

$$R = R_{ref}\left[1 + \alpha\left(T - T_{ref}\right)\right] = 235[1 + 0.00206(T - 34.3)] = 0.5124\,T[°C] + 218.4 \quad (1)$$

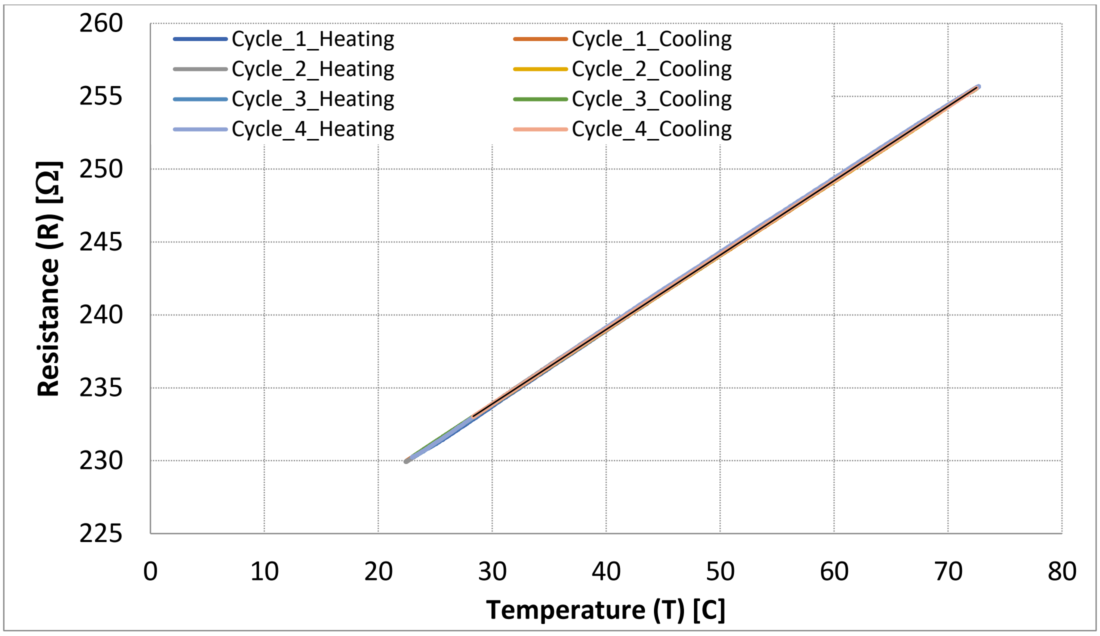

Figure 3. Microheater electrical resistivity vs. temperature – thermal calibration cycles.

II. Characterization of the coupled MH and the electronic system.

The coupling of the electronic system might alter the response time of the heater sensitivity to the power input. The pulse has three main characteristics: input voltage, power resistor value and pulse duration. We inspected each of these parameters and constructed a calibration table using power resistors that do not change their temperature as a function of the power. Within the domain that was examined, it was found that for voltage inputs ranging from 10V to 20 V, the error values span over about 1%, and are independent of the resistivity value for R ≥ 250 Ω, as seen in **Figure 4**.

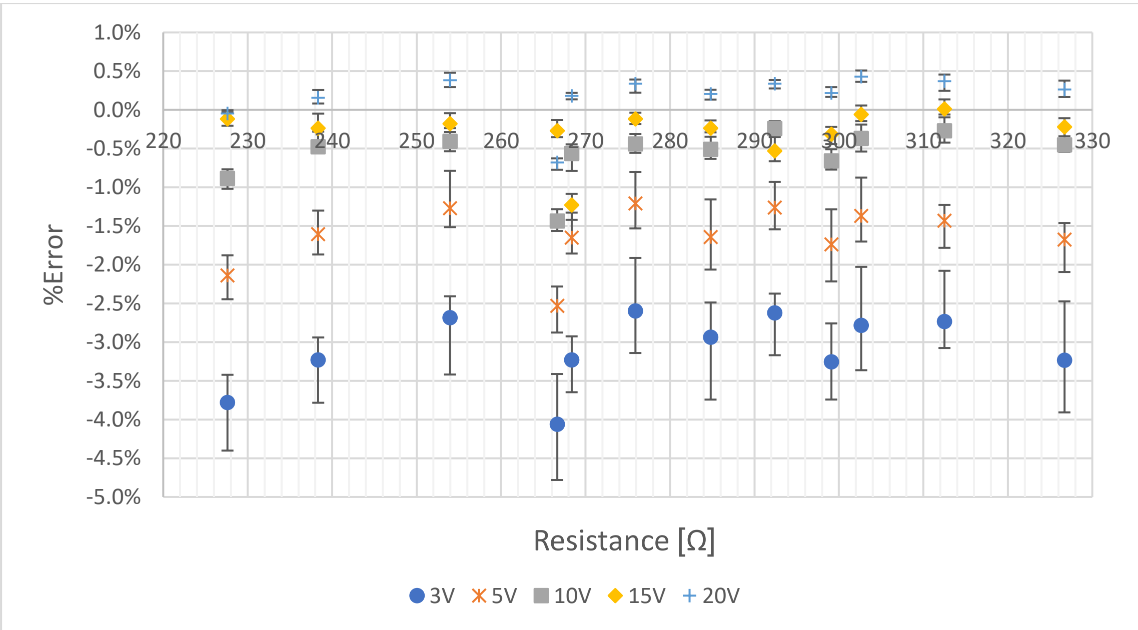

Figure 4. Thermal characterization of the micro-heater (MH).

Similar results were obtained for various pulse durations (8μs to 800μs). However, as we are concerned with rapid heating, ONB can appear after a few microseconds, depending on the power input rate into the liquid. For that reason, it is imperative to characterize the effect on the resistivity measurement during the first microseconds. For that purpose, a special code was written that enables us to construct a correction function for the fast transient processes.

Results and discussion

Experiments were conducted with HFE-7100 (3M) for a couple of reasons:

a. Our MH requires a dielectric fluid.
b. HFE-7100 is a fluid with low saturation temperature (61°C at atmospheric pressure) and relatively low latent heat of evaporation (132 kJ/kg) [13]. Compared to water, much less energy is required in order to reach nucleation conditions.
c. HFE-7x00 series is becoming an important liquid in the industry (replacing FC-72 in many cases).

During the initial period of time, until ONB occurs, transient conduction is the dominant heat transfer mechanism. This is due to the fact that the liquid is at rest initially (pool boiling) and since natural convection in a liquid cannot be developed during the short period of times used in the current study (<1 ms).

Nucleation will occur in a very thin liquid layer adjacent to the wall, in which sufficient superheating is achieved in order to form and sustain a critical nucleon. Prior to a nucleation event, the microheater is in contact solely with liquid, and the temperature of the liquid can be assumed to be equal to the surface temperature of the micro-heater. Sensible heat is dissipated in the liquid and the temperature increases monotonically yet no linearly until the nucleation occurs. At this moment some of the latent heat goes into the formation of a new phase and the temperature reaches an inflection point. After the nucleation event, the average, measured temperature increases more steeply, as the MH is now in contact with vapor as well, which has a lower thermal conductivity coefficient

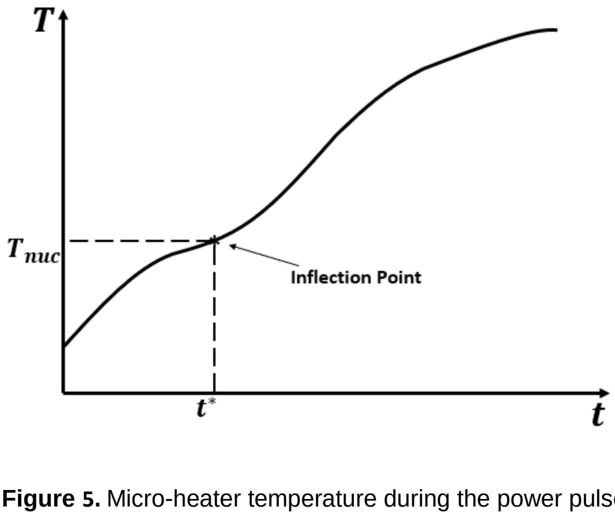

Figure 5. Micro-heater temperature during the power pulse.

To start with, we conducted experiments with an untreated liquid, initially at room temperature, which corresponds to about subcooling degree of 35°C. Namely no cleaning or degassing procedures were applied to the liquid, thus it is expected that the liquid has many dissolved gasses and impurities that serve as nucleation centres, on top of the heater layers that also serve as nucleation centres. Therefore, the superheat degree that can be attained is limited for the slow to intermediate heating rates that are presented in this study.

Figure 6 presents the input voltage (black, continuous curve) and the voltage of the MH (black, dashed curve), as illustrated in the simplified electrical gate diagram in **Figure 7**. Also presented in **Figure 6** is the inferred Micro-Heater temperature (blue), after implementing the correction function derived in the previous section. The pulse duration here is 80µs, while the rise time of our in-house electronic gate system is designed and characterized to be about 10 ns.

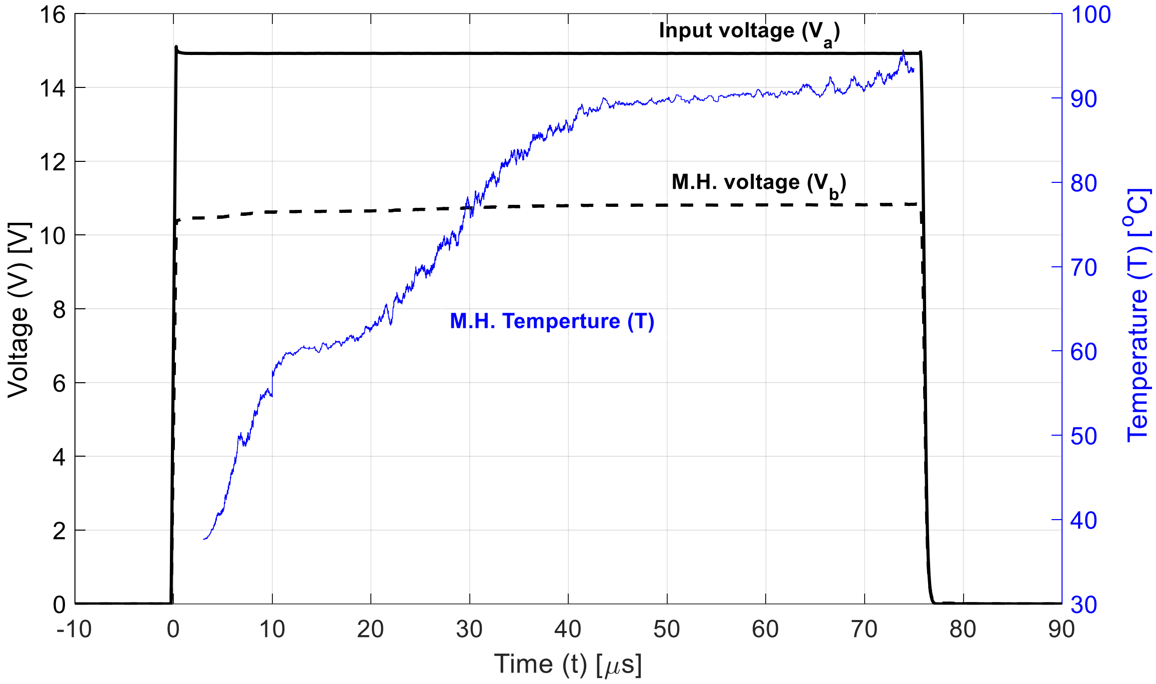

Figure 6 – Voltage measurement, and its translation to Micro-Heater resistance.

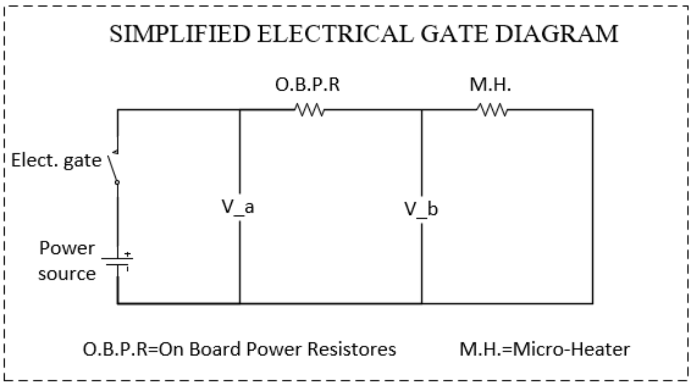

Figure 7 – Simplified electrical gate diagram.

Figure 8 presents the evolution of the power and the temperature for an input voltage of 15 V and pulse duration of 80 µs. Though the input voltage (black, continuous curve in **Figure 6**) is constant, the power increases with time mainly due to the increase in electrical resistivity of the MH, as a result of its increased temperature (see Eq. (1)). The transient conduction period is evident in the beginning of the heating process, and nucleation occurs at the inflection point at 10 µs. It can be seen that with an untreated liquid HFE-7100, nucleation occurs at the saturation temperature at the ambient pressure (60°C at 10^5 Pa). After nucleation the temperature continues to rise, as expected, since the power was still applied.

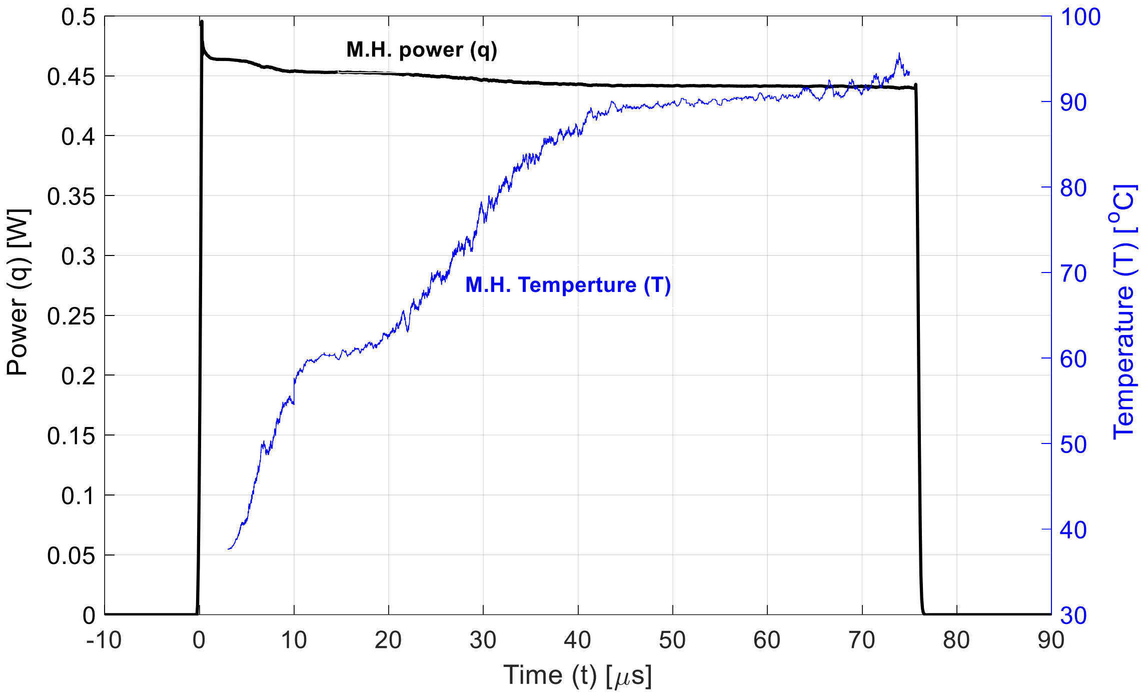

Figure 8 – Evolution of the power and the temperature for V= 15 V and pulse duration of 80 μs.

Summary and Conclusions

An experimental study was conducted to explore the response of HFE-7100 to rapid heating conditions, in light of the lack of data in the literature. A fast system was designed, realized and calibrated. The nucleation temperature was determined by measuring the change of resistance of the Pt MH during the application of a power pulse with variable durations.

The boiling temperature at the appropriate ambient pressure was achieved and deduced both by the electrical signal and the photographic study using the HSV camera.

Future work will include more rapid heating rates and a study of the subcooling effects.

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