

APPLICATION OF OPTICAL SUPERHEATING OF A SURFACE TO  
INVESTIGATION OF THE ONSET OF BOILING OF CRYOGENIC LIQUIDS

G. F. Kopytov, B. V. Novikov, and A. G. Shklyayev

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A method is described for detecting the onset of boiling of cryogenic liquids. Boiling is initiated by superheating, using an optical system, of a surface placed in a saturated cryogenic liquid. The experimental dependence of the thermal flux on the amount of surface superheating is obtained.

INTRODUCTION

Questions connected with the boiling of liquids (see, e.g., [1-5]) have a more than thirty-year history and have stimulated work on damage prevention in nuclear reactors [6]. The present work is an attempt to apply this accumulated experience to cryogenic liquids (CL).

In the vast majority of CL vessels, the liquids are in a saturated state. Their high sensitivity to increase of heat addition under these conditions suggests a simple method of detection of the onset of boiling (DOB): monitoring the increase of superheating of the liquid adjacent to a surface.

The onset of superheating of the surface over which the CL flows, of order tenths of degrees Kelvin, gives rise to a small thermal flux, of order a fraction of a watt per square centimeter. These thermal characteristics imply that heat exchange between the liquid and the solid surface is transitional from convective to boiling heat exchange [7]. An exactly similar thermodynamic pattern arises in CL storage safety.

For comparison, we have selected two methods of DOB of CL [6]: 1) measurement of pulsations in the liquid, located in the active zone, using a surface-mounted thermocouple; and 2) monitoring of the acoustic spectrum in the active zone.

With respect to the first method, the opinion is held in the literature [8] that it is impossible to use a thermocouple on a heated surface (HS) as a temperature transducer. This opinion is correct for developed, bubble-forming boiling, especially in the approximation of first burnout. At the beginning of boiling, however, the thermocouple method may be used for measurement of surface superheating for DOB.

EXPERIMENTAL

DOB of CL, as pointed out above, was accomplished by monitoring the onset of superheating of the surface on which the liquid first attains saturation [9].

To create superheating, an optical system was used which included a halogen-lamp light source, a reflector, a collecting lens, and a HS. The HS consisted of a copper ring with diameters 20 and 10 mm, with thickness 0.5 mm after lathe-turning, blackened with a mixture of soot and butvar-phenolic adhesive. The nonirradiated parts of the ring were thermally isolated.

The junctions of seven differential copper-constantan thermocouples with spherical junctions of diameter about 0.5 mm were arranged uniformly around the ring, flush with the HS. The opposite junctions were connected to the common ground. The HS with the thermocouples was placed in an optically transparent Dewar vessel. For the CL, the following were used: nitrogen, oxygen, air, argon, a propane-butane mixture, and freon-12, in the saturated state. To process the thermocouple signal, a photoelectric amplifier with input voltage stabilization was used.

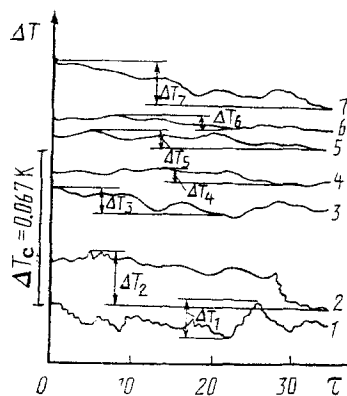


Fig. 1

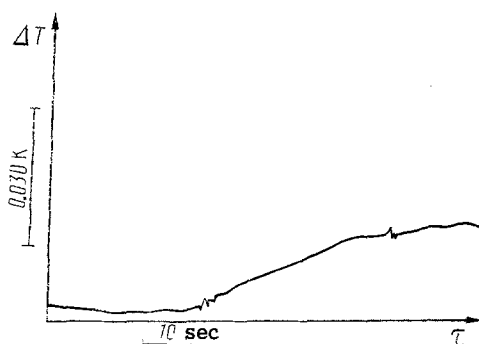


Fig. 2

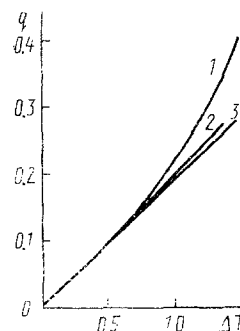


Fig. 3

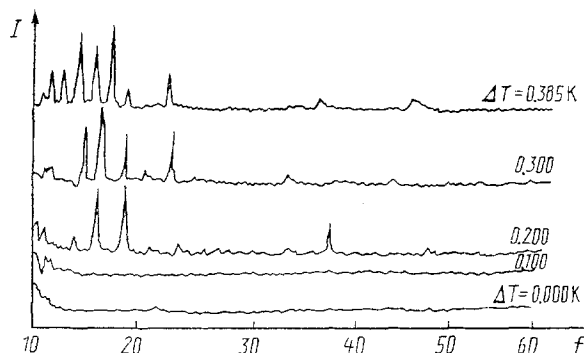


Fig. 4

Fig. 1. Oscillation of the superheating profile for a varying number of thermocouples.  $\Delta T$  is in K;  $\tau$ , in sec.

Fig. 2. Growth of the profile  $\Delta T = f(\tau)$  with increase of the heated surface superheating.

Fig. 3. Experimental dependence of the heat flux on the amount of heated surface superheating: 1) nitrogen; 2) propane; 3) freon. The heat flux  $q$  is given in  $W/cm^2$ .

Fig. 4. Experimental results of acoustic detection of the onset of boiling for nitrogen. The intensity  $I$  is given in dB, and the frequency  $f$  in kHz.

The amplifier voltage-temperature characteristic ( $\mu\text{V}$  to K calibration) was determined using two saturated liquids, liquid nitrogen of 96% purity and liquid argon of high purity. The known equilibrium temperatures are 77.348 K for nitrogen and 87.290 K for argon. Recording of the temperature difference between the two liquids with the amplifier scale in the range 0-75  $\mu\text{V}$  over a period of tens of seconds gave a spread of readings in the range of half a division, which on the scale of 75 divisions represented 0.5  $\mu\text{V}$ , corresponding to 0.03 K.

The thermal flux  $q$  from the source, which initiated the boiling, was determined from measurement of the irradiation power in  $\text{W}/\text{cm}^2$ ; the precision of this measurement was 0.025  $\text{W}/\text{cm}^2$  over the range 0-0.5  $\text{W}/\text{cm}^2$ .

It was first determined that DOB of CL using a single thermocouple is possible. Figure 1 presents, for stabilized superheating of the HS by 0.4 K, the oscillation of the superheating profile (the amount of superheating  $\Delta T$  versus the time  $\tau$ ) for various numbers  $n$  of thermocouples: curve 1 corresponds to measuring using seven thermocouples; curve 2, using six; and so on. No tendency toward increased spread of the profile  $\Delta T = f(\tau)$  with decreasing  $n$  is seen.

In the single-thermocouple experiment, the onset of CL boiling is observed for superheating of the HS.

From Fig. 2 it follows that the function  $\Delta T = f(\tau)$  increases with increasing HS superheating, reaching 0.015 K and causing boiling of nitrogen to start.

Measurements of the HS superheating corresponding to temperature differences lower than 1 K were carried out with increased amplifier sensitivity (in the range 0-7.5  $\mu\text{V}$ ), which allowed the observation of superheating of the order of hundredths of a degree Kelvin with good reproducibility.

In Fig. 3 is shown the experimental dependence of the thermal flux on the amount of HS superheating for several liquids.

In view of the extension of acoustic diagnostics to boiling [10], we have performed a comparative estimate of the sensitivity of thermocouple DOB. In Fig. 4 are shown experimental results of acoustic DOB for nitrogen in a cryostat with geometric proportion  $H \gg d$ .

Underheating of nitrogen was provided by evacuation of its vapor. A piezoelectric transducer with characteristic frequency 3 MHz served as the acoustic receiver; the signal was processed using a spectrum analyzer. The connecting cable was cooled by nitrogen vapor in a heat exchanger. The most informative frequency range, 10-50 kHz [8], was analyzed.

The occurrence of resonance peaks, corresponding to the onset of boiling of nitrogen at the HS, was noted for HS superheating of 0.2 K.

The investigation performed allows the following conclusions to be drawn:

- Single-thermocouple DOB of CL is possible;
- The obtained relations  $q = f(\Delta T)$  for the onset of boiling of CL in a large volume supplement the analogous general relation; and
- The method of thermocouple DOB of CL is an order of magnitude more sensitive than the acoustic method.

#### NOTATION

$T_C$ , boiling temperature of a cryogenic liquid, K;  $\tau$ , time, sec;  $n$ , number of thermocouples;  $\Delta T_C$ , amount of superheating of the heated surface in relation to the boiling temperature of the corresponding cryogenic liquid, K;  $q$ , thermal flux,  $\text{W}/\text{cm}^2$ ;  $H$ , cryostat height, mm;  $d$ , cryostat interior diameter, mm.

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## VAPORIZATION IN SUPERHEATED OLIGOMER AND POLYMER LIQUIDS

E. D. Nikitin and N. V. Bessonova

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The method of impulsive heating of a wire probe is used to measure the spontaneous boiling point of commercial synthetic isoprene rubber and its solutions in saturated hydrocarbons, polyethylene glycols, and polyethylsiloxanes with allowance for thermal degradation of the polymer. A formula is obtained to describe the dependence of the boiling point of the polymer solution on heating time.

The boiling of solutions of polymers is characterized by certain features which are reflective of their properties - high viscosity, the presence of stratified regions, and thermal degradation at high temperatures. In the present study, we will focus on the boiling of concentrated solutions undergoing rapid heating - when the polymer component undergoes thermal degradation. A survey of studies of the steady boiling of dilute polymer solutions was presented in [1], while data on the boiling of certain stratifying polymer solutions was reported in [2, 3].

Vaporization in pure liquid polymers cannot occur without their thermal degradation, since high-molecular-weight compounds are nearly incapable of being converted to the gaseous state. With an increase in the degree of polymerization, it also becomes increasingly difficult for a macromolecule to undergo conversion from the condensed phase to the gas phase; on the other hand, an increase in the length of the chain is generally accompanied by a decrease in the effective activation energy or thermal degradation [4]. Thus, there is a certain maximum molecular weight at which vaporization is still possible. This limit, of course, depends on the structure of the polymer. For polyethylene, for example, this limiting value is of the order of 540 [5]. Tests that we conducted with commercial polyethylsiloxanes [6] - for which boiling is the same as for low-molecular-weight liquids - showed that this limit is no lower than 1300-2000 for the oligomers we studied. It must be noted, however, that special methods make it possible to obtain molecules with a weight up to  $6 \cdot 10^6$  in the gas phase [7].

As a result, if a liquid polymer is heated, it first undergoes thermal degradation. Then the products of this process dissolve in the liquid phase. Finally, bubbles filled with degradation products begin to appear. This pattern may be complicated by further decomposition of the liquid into several coexisting liquid phases and their subsequent boiling.

Let us suppose that at the initial moment of time a polymer solution has a concentration  $c_0$  and temperature  $T_0$  which is less than the temperature at which thermal degradation begins. Pressure in the solution then remains constant, while its temperature increases with

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Institute of Thermophysics, Russian Academy of Sciences, Ekaterinburg. Translated from *Inzhenerno-fizicheskii Zhurnal*, Vol. 62, No. 2, pp. 271-275, February, 1992. Original article submitted July 9, 1991.