

## Temperature and Concentration Relaxation in Phase-separated Superfluid $^3\text{He}$ – $^4\text{He}$ Mixtures

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*The kinetics of the temperature and concentration variations in the superfluid  $^3\text{He}$ – $^4\text{He}$  mixtures with initial concentration of 9.8%  $^3\text{He}$ , and heated from below, was studied experimentally under the pressure of 0.38 bar over a temperature range of 150–400 mK. It is found that in contrast to homogeneous liquids, the temperature and concentration relaxation in phase-separated mixtures can be described by a superposition of two exponential processes in which the time constants of temperature and concentration variations coincide. If the initial mixture was homogeneous and phase separation was triggered by a heat flow, the temperature and concentration vary non-monotonically and exhibit anomalous features at the moment of phase separation. In this case the phase transition starts in the metastable superfluid, formed out of a quite supersaturated mixture where the nucleation of the new phase may be caused by quantized vortices. The results are analyzed in terms of two possible mechanisms of relaxation—the acoustic mechanism with the second sound velocity and the diffusive one connected with dissipative flows of impurity and thermal excitations. It is shown that the measured relaxation times agree with a prediction of the theory.*

**KEY WORDS:** nucleation; phase separation; relaxation; superfluid  $^3\text{He}$ – $^4\text{He}$  mixture.

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## 1. INTRODUCTION

The processes of temperature and concentration relaxation in  $^3\text{He}$ – $^4\text{He}$  superfluid mixtures are rather specific.<sup>8</sup> A heat flow applied to the system induces temperature and concentration gradients which appear simultaneously and thus the processes of thermal conductivity, thermal and mass diffusion are interrelated in these mixtures. According to theoretical studies of the kinetic and relaxation processes in superfluid mixtures,<sup>6,5</sup> the temperature and concentration relaxation in this system is determined by both the second sound mechanism and the dissipative diffusion.

The first systematic experimental research of the processes of temperature and concentration relaxation in  $^3\text{He}$ – $^4\text{He}$  mixtures (51–67%  $^3\text{He}$ ), was made by Behringer and Meyer<sup>2,3</sup> at rather high temperature (0.8–1.2 K). The authors showed that in the superfluid region the relaxation times of temperature and concentration coincide and agree with the calculation based on the hydrodynamic theory for solutions.

It is natural that the relaxation processes in superfluid  $^3\text{He}$ – $^4\text{He}$  mixtures should exhibit new peculiarities at lower temperatures where first the roton and then the phonon contribution becomes negligible, and the impurity excitations start to control all kinetic processes. In this case, mixtures with concentrations over 6.7% of  $^3\text{He}$  separate into the superfluid and normal phases. The temperature and concentration relaxation in  $^3\text{He}$ – $^4\text{He}$  mixtures at low temperatures was considered theoretically by solving the kinetic equation for impurity excitation gas in superfluid mixtures.<sup>1,10</sup> The first experimental investigations<sup>15</sup> of temperature and concentration relaxation in concentrated superfluid  $^3\text{He}$ – $^4\text{He}$  mixtures at low temperatures detected new features of the processes that appear due to phase separation. The present study is a continuation of the work in Ref. 15 and is concerned with a detailed investigation of temperature and concentration relaxation with particular attention given to the region of phase separation.

## 2. EXPERIMENTAL

The kinetics of the temperature and concentration variations in a superfluid  $^3\text{He}$ – $^4\text{He}$  mixture was measured in the presence and absence of a heat flow. The experimental cell is similar to that used earlier<sup>15–17</sup> in the measurements of effective thermal conductivity, thermal stability, relaxation, and dissipation processes in superfluid  $^3\text{He}$ – $^4\text{He}$  mixtures, but it differs in the size (29.5 mm long and 24 mm in diameter) and in the arrangement of temperature and concentration gauges. These innovations have enabled registration of finer kinetic effects. The heat flow was produced by a heater (Fig. 1) wound onto the lower cap of a cylindrical

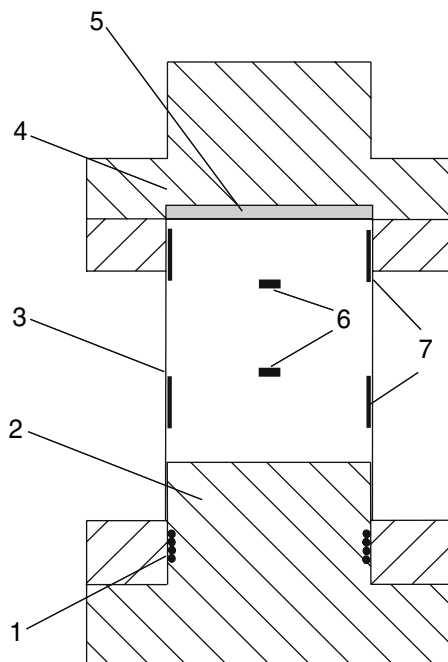


Fig. 1. Schematic of the measuring cell: 1 – heater; 2 – bottom flange; 3 – cylindrical wall; 4 – top cap; 5 – heat exchanger; 6 – thermometers of resistance; 7 – capacitance gauges for concentration measurement.

measuring cell. The polished flat face of the cap, mounted at the cell bottom, was in contact with the mixture under investigation. The cylindrical cell wall was made of thin-sheet stainless steel. The cap at the top of the cell was in permanent thermal contact with the mixing chamber. The heat exchanger was made of sintered ultra fine Ag powder mounted on the cap, its area being about  $1\text{ m}^2$ .

The design of the capacitance sensors for concentration measurement was changed too. The sensors are cylindrical single-layer coils with a diameter slightly smaller than the inner diameter of the cell. The windings were made out of two  $100\text{ }\mu\text{m}$  copper wires. The values of concentration variations in the mixture were calculated from the change in the capacitance between these wires using the Clausius–Mossotti relation. The gauges were spaced in the cell  $11.2\text{ mm}$  apart but arranged to remain both in the diluted phase even after the phase separation. The upper capacitance gauge was placed at  $1\text{ mm}$  distance from the heat exchanger.

The nominal capacitance of the sensors was 70 pF and the relative error of the capacitance measurement was  $10^{-5}$ .

The temperature was measured with two miniature RuO<sub>2</sub> resistance thermometers fixed at distances of 9.3 and 20.5 mm from the lower end of the cell. The thermometers were calibrated against a <sup>3</sup>He melting curve thermometer. The absolute error of the temperature measurement was 0.5 mK. The thermometer readings were registered automatically with 3-s intervals (cf. 1-minute intervals in Refs. 15–17), which enabled us to resolve the fast processes connected with the phase separation. The temperature of the upper flange of the cell was kept constant throughout the experiment. The specific power fed to the heater varied from 0.5 to 25  $\mu$ W/cm<sup>2</sup>. The temperature and concentration relaxations were measured by switching the heat flow on and off. The mixture with an initial concentration of 9.8% <sup>3</sup>He was investigated over a temperature interval of 150–400 mK under a pressure of 0.38 bar.

### 3. RESULTS AND DISCUSSION

The experimental results show that the temperature and concentration relaxation in the superfluid <sup>3</sup>He–<sup>4</sup>He mixture is essentially dependent on whether or not the phase separation occurs in the mixture. When no heat flow was applied, the temperature of the phase separation at the pressure of 0.38 bar was  $T_s = 210$  mK. On heating the mixture from below, the <sup>3</sup>He concentration increased in the upper colder region of the cell, which initiated phase separation in this region at higher temperature.<sup>15</sup> It was shown in Ref. 18 that the phase interface is a destabilizing factor that can cause thermal instability. Since the interfacial tension coefficient depends on temperature and concentration, any disturbance of the interface must generate a tangential force directed along the surface tension gradient. This produces a radial liquid flow along the interface and can lead to instability. For this reason it is essential to keep the top cap parallel to the interface. While planning this experiment, special attention was focused on adjustment of the cell. Since temperature and concentration relaxations are significantly influenced by phase separation, the experimental results can be subdivided into three groups according to the experimental conditions:

Regime 1: The temperature of the cell is rather high and no phase separation occurs even when applying a heat flow, i.e., relaxation proceeds in a homogeneous mixture.

Regime 2: The starting temperature of the cell (no heat flow) is lower than the phase separation temperature ( $T \leq T_s$ ), i.e., a heat flow is applied to the mixture with separated phases.

Regime 3: Phase separation starts after the heat flow is switched on, i.e., it occurs during temperature and concentration relaxation.

#### 4. RELAXATION IN HOMOGENEOUS MIXTURE

Above the temperature of phase separation, where the superfluid  $^3\text{He}$ - $^4\text{He}$  mixture is homogeneous, the temperature and concentration relaxation follows the exponential law (see Ref. 15). In present study, special attention was focused on comparing the relaxation processes in the presence and absence of a heat flow, in particular the temperature difference  $\Delta T = T_1 - T_2$  between two thermometers was studied. The experimental results show that after switching on the heat flow, the variation  $\Delta T(t)$  can be described by the time dependence:

$$\Delta T(t) = \Delta T_M [1 - \exp(-t/\tau_T)], \quad (1)$$

where  $\Delta T_M$  is the maximum difference  $\Delta T(t)$  between the readings of the thermometers, and  $\tau_T$  is the time constant of the temperature relaxation. At switching off the heat flow,  $\Delta T(t)$  is:

$$\Delta T(t) = \Delta T_M \exp(-t/\tau_T). \quad (2)$$

Figure 2 shows the time dependences  $\Theta_1(t) = 1 - \Delta T(t)/\Delta T_M$  (heat flow on) and  $\Theta_2(t) = \Delta T(t)/\Delta T_M$  (heat flow off) at the flow density  $Q = 1.5 \mu\text{W/cm}^2$  and starting temperature  $T = 265 \text{ mK}$ . It is seen from Fig. 2 that the temperature kinetics in a homogeneous mixture is the same at switching a heat flow on or off. The time constant  $\tau_T = 640 \pm 70 \text{ s}$  of temperature relaxation is practically independent of  $Q$ . This value is in good agreement with the results of Ref. 15 within the accuracy of the experiment. As the temperature varies, the relaxation of concentration in a homogeneous mixture follows the same exponential law (see above). The time constants  $\tau_x$  and  $\tau_T$  are practically equal irrespective of whether the heat flow is switched on or off.

#### 5. RELAXATION IN SEPARATED MIXTURE

When the temperature of the mixture was rather low and the phase separation occurred before switching on a heat flow, the relaxation of the temperature difference  $\Delta T$  between two thermometers after applying a heat flow cannot be described adequately only with a single exponential function. In this case, the time dependence of  $\Delta T(t)$  can be approximated by a superposition of two exponentials:

$$\Delta T(t) = \Delta T_M [1 - \alpha \exp(-t/\tau_{T1}) - \beta \exp(-t/\tau_{T2})] \quad (3)$$

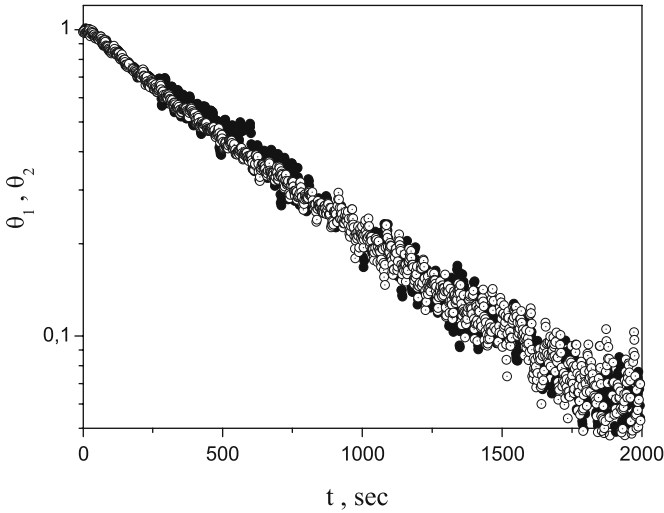


Fig. 2. Typical behavior of temperature variation in a homogeneous mixture of  $^3\text{He}$ - $^4\text{He}$  after switching heat flow on (●) and off (○) (see the text).

with two time constants  $\tau_{T1}$  and  $\tau_{T2}$  and two weight factors  $\alpha$  and  $\beta$ . One relaxation process is characterized by the time  $\tau_{T1} = 630 \pm 70$  s, which nearly coincides with  $\tau_T$  for a homogeneous mixture. This process is determined by the effective thermal conductivity of the superfluid mixture. The other relaxation process is faster ( $\tau_{T2} = 160 \pm 70$  s) and is presumably connected with the change in the temperature gradient induced by the heat flow. This change in turn affects the volume ratio between the separated phases, which provokes release of extra heat of the phase transition.

After switching off the heat flow, the relaxation of the temperature is also described by two exponentials:

$$\Delta T(t) = \Delta T_M [\alpha \exp(-t/\tau_{T1}^*) - \beta \exp(-t/\tau_{T2}^*)]. \quad (4)$$

However, in contrast to a homogeneous mixture  $\tau_{T1}^*$  and  $\tau_{T2}^*$ , the time constants of this process do not coincide with the corresponding times of the heat-on mode.

It is convenient to describe the kinetics of temperature at switching the heat flow on and off as time dependences of the parameters that were introduced for a homogeneous mixture in accordance with Eq. (3). The data in Fig. 3 ( $T = 180$  mK,  $Q = 14.5 \mu\text{W}/\text{cm}^2$ ) shows the features of the temperature relaxation in separated mixtures. The concentration  $x(t)$  of the mixture in the heat-on mode was measured with the lower

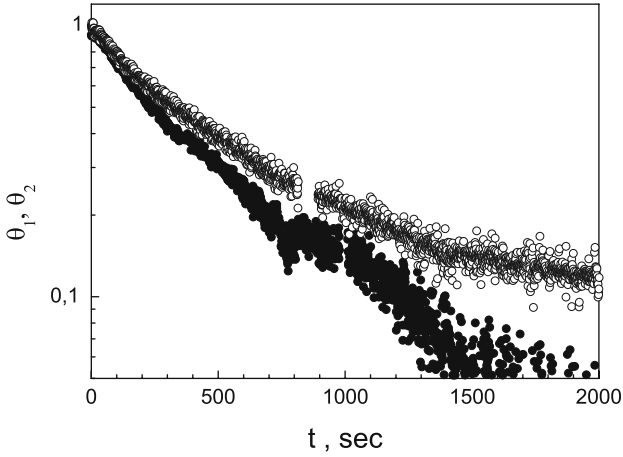


Fig. 3. Typical relaxation of temperature in a separated mixture after switching heat flow on ( $\bullet$ ) and off ( $\circ$ ).

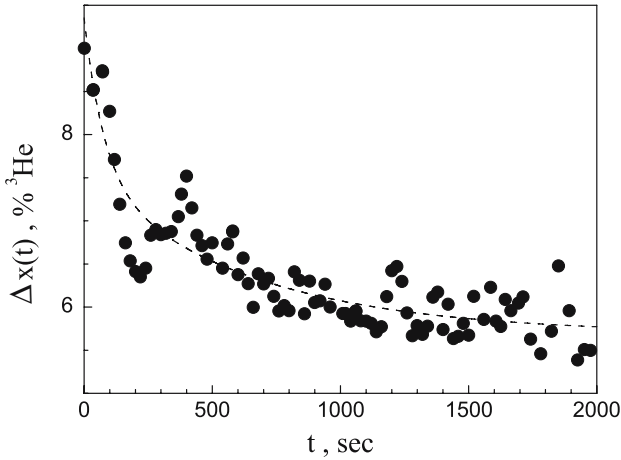


Fig. 4. Time dependence of the concentration variation in a mixture, registered by the bottom gauge after switching the heat flow on.

concentration gauge and the results are shown in Fig. 4, for the same  $T$  and  $Q$  as in Fig. 3. When a heat flow is applied, the concentration of the superfluid mixture decreases in the lower part of the cell by a value  $\Delta x(t)$ :

$$x(t) = x_0 - \Delta x(t) \quad (5)$$

( $x_0$  is the initial concentration), because  $^3\text{He}$  moves with the normal component to the upper (colder) part of the cell. The  $\Delta x(t)$  can be written as a dependence totally identical to that of Eq. (4):

$$\Delta x(t) = \Delta x_M [1 - \alpha \exp(-t/\tau_{X1}) - \beta \exp(-t/\tau_{X2})], \quad (6)$$

where  $\Delta x_M$  is the maximum change in the concentration under acting of the heat flow;  $\tau_{X1}$  and  $\tau_{X2}$  are the time constants of the concentration relaxation. It is seen that like in a homogeneous mixture,  $\tau_{X1}$  and  $\tau_{X2}$  in the heat-on mode are close to the time constants of temperature relaxation  $\tau_{T1}$  and  $\tau_{T2}$ .

## 6. TEMPERATURE AND CONCENTRATION RELAXATION DURING PHASE SEPARATION

The starting mixture was a homogeneous liquid at the temperature above the temperature of the phase separation. Figure 5 shows the changes in the temperature (Fig. 5a) and the concentration (Fig. 5b) registered with the thermometers and the concentration sensors after switching on the heat flow. Although the thermometers and the concentration sensors were fixed at a rather long distance from one another, they registered the moment of switching on the heat flow simultaneously. The fast relaxation of temperature and concentration throughout the liquid is due to the acoustic mechanism of relaxation at the second sound velocity. In our experiment the relaxation time was  $10^{-2}\text{s}$ . Unlike pure  $^4\text{He}$  in which steady gradients of temperature are impossible because second sound mechanism, in superfluid mixtures temperature and concentration gradients can be produced simultaneously, and their relaxation has the same velocities in the acoustic and diffusive modes. The acoustic relaxation is very fast and cannot be registered in this experiment straight forwardly. The changes in the temperature and the concentration shown in Fig. 5a, b (after arrow 1) correspond to the relaxation caused by the diffusive dissipative flow of impurities and thermal excitations.

In this region  $T(t)$  and  $x(t)$  are smooth functions of time and free of anomalies. They can be approximated by one exponential for which the time constants  $\tau_T$  and  $\tau_x$  are equal and determined by the effective thermal conductivity of the mixture. The time constants  $\tau = \tau_T = \tau_x$ , obtained as a function of the average temperature of the cell, are shown in Fig. 6 in which the dashed line corresponds  $\tau$ -values calculated from the kinetic theory for a system of thermal and impurity-induced excitations, taking into account the boundary conditions. The relaxation time  $\tau$  in a superfluid solution can be presented as Ref. 5.



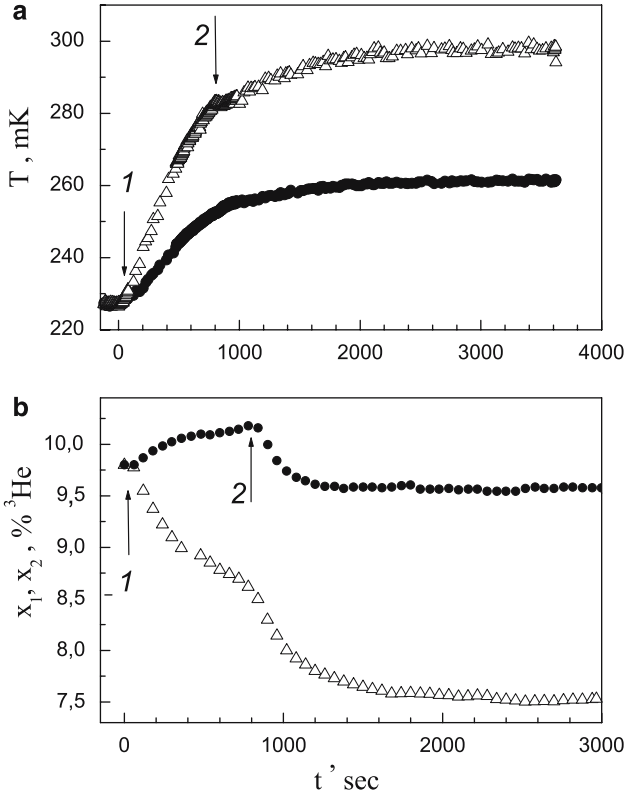


Fig. 5. Temperature (a) and concentration (b) variations registered by the top (●) and bottom (Δ) gauges after switching on a heat flow of  $8.3 \mu\text{W}/\text{cm}^2$ . The initial temperature is 228 mK. Arrow 1 corresponds to switching on the heat flow and arrow 2 to the beginning of the phase separation.

$$\tau = \frac{h^2 \rho C_{P,x}}{\kappa_{\text{eff}}} \frac{1 + A^*}{B^2(A^*)}, \quad (7)$$

where  $h$  is the measuring cell height,  $\rho$  the solution density,  $C_{P,x}$  the heat capacity,  $\kappa_{\text{eff}}$  the effective thermal conductivity coefficient,  $A^*$  is the thermodynamic parameter determined by the properties of the elementary excitation gas:

$$A^* = (k_T^*)^2 \left( \frac{\partial \mu}{\partial x} \right)_{T,P} \frac{1}{T \cdot C_{P,x}}. \quad (8)$$

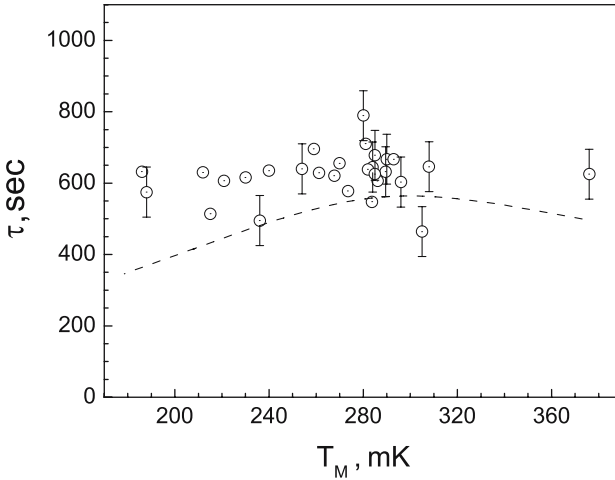


Fig. 6. Temperature dependence of the time constant of temperature relaxation in a superfluid solution. Dashed line (-) is calculated from Eq. (7).

In the low-temperature limit when the properties of the solution are determined only by the impurity-induced excitations,  $A^* \rightarrow 0$ . In the other limiting case the thermal excitations are dominant and  $A^* \rightarrow \infty$ . In the intermediate interval  $A^*$  characterizes the relation between the contributions of the thermal and impurity-induced excitations to the kinetic properties of the solution. In Eq. (8)  $k_T^*$  is the thermodiffusion ratio of the solution

$$k_T^* = -T \frac{\nabla x}{\nabla T} \quad (9)$$

determining the correlation between the heat and mass diffusion in the solution. The value of  $\mu$  is equal to the difference between the chemical potentials of  $^3\text{He}$  and  $^4\text{He}$  in the solution  $\mu = \mu_3 - \mu_4$ . The derivative  $(\partial\mu/\partial x)_{T,P}$  is commonly called the concentration susceptibility. The parameter  $B$  in Eq. (7) is found as a solution of a transcendental equation in Ref. 3:

$$\frac{\tan B}{B} = -\frac{1}{A^*}. \quad (10)$$

Note that the parameter  $A^*$  can be expressed (see Refs. 1,10,11) in terms of the hydrodynamic parameters  $u_{2\varepsilon}^2$  and  $u_{2N}^2$ :

$$A^* = \frac{u_{2\varepsilon}^2}{u_{2N}^2}, \quad (11)$$

$$u_{2\varepsilon}^2 = \frac{\rho_s}{\rho_n} \frac{\tilde{s}^2 T}{C_V}, \quad \tilde{s} = s - x \frac{\partial s}{\partial x}, \quad (12)$$

$$u_{2N}^2 = \frac{\rho_s}{\rho_n} \frac{x}{\rho} \left( \frac{\partial P_f}{\partial x} \right)_T, \quad (13)$$

where  $s$  is the solution entropy,  $P_f$  the osmotic pressure of the impurity-induced excitations, and  $u_{2\varepsilon}^2$  and  $u_{2N}^2$  are connected with the second sound velocity  $u_2$  in the solution by the simple ratio:

$$u_2^2 = u_{2N}^2 + u_{2\varepsilon}^2. \quad (14)$$

This approach is convenient because Eq. (14) makes it possible to test independently the validity of the theory by comparing the calculated and experimental second sound velocities in the solution. To compare the experimental and calculated relaxation times  $\tau$ , we first estimated the parameter  $A^*(T)$  by Eqs. (12) and (13). The values of  $s$ ,  $P_f$ , and  $C_V$  were taken from Ref. 12. In our experiment  $A^*$  varied within 0.6–0.7. Then  $\tau$  was calculated from Eqs. (7) and (10) using  $\kappa_{\text{eff}}$  obtained from stationary measurements (this experiment). The calculated  $\tau$  is shown in Fig. 6 (dashed line) along with constant time values. Above 220 mK experiment and theory are in good agreement.

After switching on the heat flow the  $^3\text{He}$  concentration in the colder upper part of the cell rises above the average value and it decreases in the lower part of the cell. Then, at a certain moment (arrow 2, Fig. 5) anomalies appear in all the curves indicating phase separation in the mixture. The concentration and temperature gauges located in different places of the cell register the phase transition in the mixture practically simultaneously. In this case the fast relaxation of the temperature and concentration is due to the second sound generated by local changes in the concentration. The concentration gauges register a sharp drop of the concentration at the moment 2 (especially in the vicinity of the upper gauge), which is connected with the formation of the concentrated phase in the upper part of the cell.

The temperature anomalies are shown on a larger scale in Fig. 7 showing the relative temperature variations  $\delta T / \delta T_{\text{MAX}}$  ( $\delta T_{\text{MAX}}$  is the maximum change in the temperature from the moment of switching on the heat flow up to the steady-state phase separation). Both thermometers exhibit a slight local cooling of the mixture at the moment of phase separation. The experimental data available are inadequate to explain unambiguously this temperature behavior.

The change in the concentration of the solution caused by the heat flow shows that a superfluid metastable mixture is formed in the cold

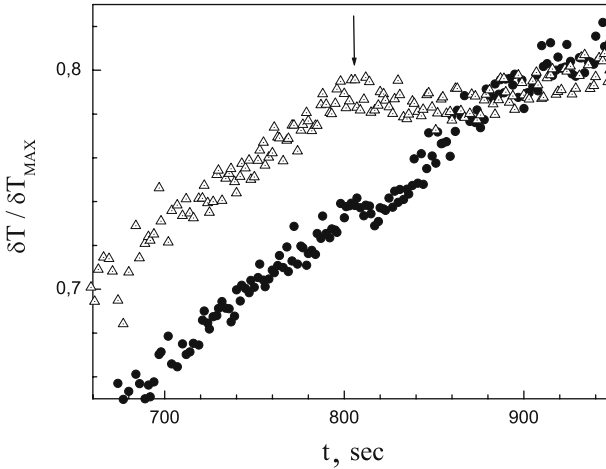


Fig. 7. Kinetics of the relative change of temperature in the vicinity of phase separation. Full circles are data registered by the bottom thermometer, triangles by the top thermometer. The arrow corresponds to the beginning of the phase separation.

part of the cell. The supersaturation of the mixture before the onset of separation was about 0.6%  $^3\text{He}$ , and the rate of the concentration variation in the metastable mixture was  $6.6 \cdot 10^{-5}\% \text{ } ^3\text{He/s}$ . This is close to the values registered in the previous experiments on a continuous variation of concentration.<sup>9</sup> This system can be convenient for investigating the nucleation kinetics in supersaturated mixtures. In this context it is natural to assume that nucleation in supersaturated mixtures is of vortex origin because the heat flow easily generates quantum vortices in He II which initiate phase separation in the supersaturated metastable supersaturated mixture.<sup>4</sup> Since the  $^3\text{He}$  atoms are drawn into the core of the vortex,<sup>13</sup> at a certain critical temperature the radius of the vortex core can grow without limit,<sup>7</sup> which means that the energy barrier has been overcome and a stable nucleus of the new phase has appeared. This initiates a phase transition. When superfluid mixtures are heated at the bottom (Refs. 17, 19), the system becomes thermally unstable if the temperature gradient exceeds a certain critical value  $\Delta T_c$ . At  $\Delta T > \Delta T_c$  the temperature gradient is no longer linearly dependent on the heat flow power. The effective thermal conductivity of the mixture increases.<sup>18</sup> It is natural to attribute the instability to quantized vortices.<sup>14</sup> The analysis of the temperature and concentration kinetics shows that after switching on the heat flow, a supersaturated metastable superfluid mixture is formed in the upper part of the cell. Its lifetime was about 500–600 s. Then two practically simultaneous

events occur: vortex instability and phase separation. The measurement error of this experiment did not permit us to find out which event started first.

The experimental data in Figs. 5–7 shown kinetic processes that appear in a homogeneous solution when heat flow is applied. In this case, the phase separation is caused by the heat flow. The reverse process – homogenization of the separated solution – occurs when the heat flow is switched off. The process does not induce significant anomalies in the temperature and concentration relaxation. The experiments show that relaxation always follows exponential law.

## 7. CONCLUSION

The series of experiments performed show that the processes of temperature and concentration relaxation, stimulated by a heat flow in  $^3\text{He}$ – $^4\text{He}$  mixtures, are characterized by certain distinctive features. The applied heat flow initiates a metastable superfluid mixture in the cold part of the cell, which exhibits considerable supersaturation. This property can be used as a tool of obtaining a metastable phase and for studying its nucleation. The phase separation of the supersaturated mixture can be caused by the formation of quantized vortices.

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