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## Surface Tension, Oil Level and Density Measurement of Oil/Refrigerant Mixture by Maximum Bubble Pressure Method

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### ABSTRACT

Refrigeration oil is used for lubrication of sliding parts and sealing of compression chambers in refrigerant compressors. The refrigeration oil generally has solubility with refrigerant. Since property of the oil such as viscosity significantly changes with dissolution of refrigerant into the oil, it is important to monitor the refrigerant concentration in the oil/refrigerant mixture in the compressor shell. Surface tension is one of the properties which change according to the refrigerant concentration in the mixture and was proposed as an indicator to measure the refrigerant concentration at high temperature and pressure condition. In this study, the surface tension was measured by a maximum bubble pressure method. A Peltier pump was developed as a bubble generation pump for the maximum bubble pressure method. Peltier element was used to increase the pressure inside a copper container by heating it with applying electric current to the elements. The pump has no moving parts, easily controls the pressure by regulating the electric current, and generates bubbles steadily at capillary tubes immersed in the mixture. A 3-capillary method, which uses three capillaries of different immersed depths and inner diameters, can measure the surface tension, oil level and density of the mixture simultaneously. The 3-capillary method is applicable to the refrigerant compressors and it allows us to monitor the oil level and the surface tension of the mixture in the compressor shell. It was found that the influence of the density of mixture on the surface tension measurement is very small in the maximum bubble pressure method. The measurement accuracy of the oil level was  $\pm 10\%$  by the 3-capillary method, and it has a feasibility to monitor the oil level in the compressor shell. A correlation between the surface tension of PVE/R410A mixture and the refrigerant concentration in oil was proposed.

### 1. INTRODUCTION

Refrigeration oil is used in refrigerant compressors for lubrication of sliding parts, sealing of leakage through clearances in compression chambers, cooling and cleaning the inside of the compressor. Some of the refrigeration oil becomes oil mist in a compressor shell and is discharged from the compressor with refrigerant to refrigeration cycle. The discharged oil must return to the compressor through the cycle to prevent an oil shortage in the compressor shell. In addition, a two-phase separation of oil and refrigerant in the compressor shell will cause a lubrication failure. For these reasons, the refrigeration oil generally has solubility with the refrigerant. Since viscosity of the refrigeration oil decreases significantly as the refrigerant dissolves into the oil, it is important to monitor the refrigerant concentration in the oil/refrigerant mixture as well as the oil level in the shell for ensuring proper lubricating condition in the compressor.

The sampling method is used to measure the refrigerant concentration by subtracting the oil from the compressor. It reduces amount of oil in the compressor and takes long time to measure. Therefore, an in-situ measurement of the refrigerant concentration in the oil stored in the compressor shell is desirable.

Properties of the oil/refrigerant mixture such as viscosity, density, capacitance, refractive index and ultrasonic vary according to the refrigerant concentration, and surface tension is also one of the properties which changes with the refrigerant concentration. There are some methods to measure the surface tension. A Wilhelmy method is often used to measure the surface tension under atmospheric pressure. Jensen and Jackman (1984) investigated the surface tension of an R113/oil mixture with several viscous mineral oils by using a ring method. Zhelezny *et al.*(2007) and Duan *et al.*(2018) measured the surface tension of the oil/refrigerant mixture at high refrigerant concentration under high temperature and high pressure environments using a capillary-rise method. Seeton and Hrnjak (2006) measured the surface tension of a POE32/CO<sub>2</sub> mixture by a maximum bubble pressure method (MBPM) with changing the temperature and concentration of dissolved CO<sub>2</sub>. In their measurement by the MBPM, two capillaries having different radii were used so that the measurement of immersed depth of capillary tips in the mixture was unnecessary. They showed that the surface tension of the mixture decreases with temperature and the mass fraction of CO<sub>2</sub>. Fukuta *et al.* measured the surface tension of PAG/CO<sub>2</sub> mixture by the MBPM (2017) and by a pendant drop method (2020) under high pressure and temperature condition. It was found that the surface tension of PAG/CO<sub>2</sub> mixture sharply decreases with an increase of the refrigerant concentration in the mixture. Although there are some studies on the surface tension of oil/refrigerant mixture, the data is not enough yet.

In this study, the surface tension of polyvinyl ether (PVE) oil/R410A mixture was measured by the MBPM. A Peltier pump was developed as a bubble generation pump for the MBPM using a Peltier element to increase the pressure inside a copper container by heating it with applying electric current to the elements. A 3-capillary method, which uses three capillaries of different immersed depths and inner diameters, was examined to measure the surface tension, oil level and density of the mixture simultaneously for the in-situ measurement of the surface tension of oil and oil level in the refrigerant compressors. A correlation between the surface tension of PVE/R410A mixture and the refrigerant concentration in oil was proposed.

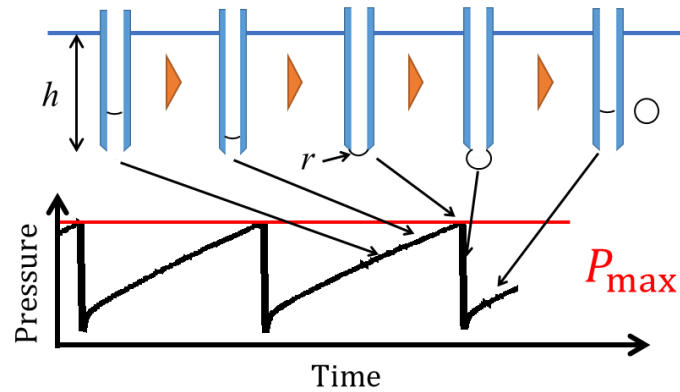
## 2. EXPERIMENT

### 2.1 Maximum bubble pressure method

The inside of a high pressure shell of actual working compressors is under high pressure and temperature condition. The maximum bubble pressure method (MBPM) is applicable to measure the surface tension of the oil/refrigerant mixture under such condition. Figure 1 shows the principle of the MBPM. A bubble is formed at a tip of a capillary tube immersed in liquid with supplying gas through the capillary tube. The bubble generation cycle is repeated when the gas is supplied continuously to the capillary. As the bubble is developed at the tip of the capillary, the curvature of the bubble decreases and the pressure inside the bubble increases. The pressure attains its maximum when the radius of the bubble becomes equal to the inner radius of the capillary tube. Based on the Young–Laplace equation, the surface tension of the liquid,  $\gamma$ , is calculated by Equation (1) under static condition.

$$\gamma = f \frac{r}{2} \left\{ P_{max} - \left( h + \frac{2}{3} r \right) \Delta \rho g \right\} \quad (1)$$

where,  $r$  is the inner radius of the capillary tube,  $P_{max}$  is the maximum pressure difference between inside the capillary tube and the atmosphere of the liquid phase,  $h$  is the immersed depth of the capillary tube in the liquid,  $\Delta \rho$  is the density difference between the liquid and the gas, and  $g$  is the gravitational acceleration.  $f$  is a correction factor for non-spherical bubble shape and found to be 0.99 in this study (Fainerman and Miller, 2004).



**Figure 1:** Pressure change in bubble formation process

### 2.2 3-capillary method

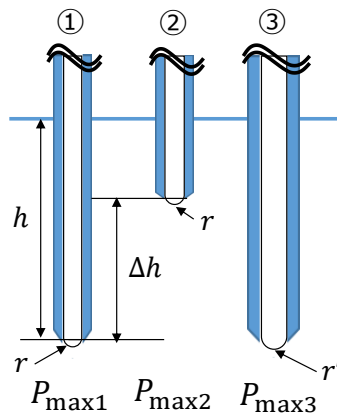
The measurement method using three capillaries in the application of the MBPM is called the 3-capillary method in this study. The 3-capillary method can measure density, capillary depth, and surface tension of a test substance simultaneously. Figure 2 shows the schematic of 3-capillary method. There is two capillaries, #1 and #2, which are same inner diameter but different in immersed depth of the capillary. The capillaries #1 and #3 are the same immersed depth but have different inner diameters of the capillary. From the maximum bubble pressures of  $P_{max1}$ ,  $P_{max2}$  and  $P_{max3}$  at each capillary, the density, the capillary depth, and the surface tension of the substance are obtained by Equations (2), (3), and (4), respectively.

$$\rho = \frac{P_{max1} - P_{max2}}{\Delta h \cdot g} \quad (2)$$

$$h = \frac{P_{max1}r - P_{max3}r'}{(r-r')\rho g} - \frac{2(r+r')}{3} \quad (3)$$

$$\gamma = \frac{r}{2} \left\{ P_{max1} - \rho g \left( h + \frac{2}{3}r \right) \right\} \quad (4)$$

where,  $\rho$  is the density,  $h$  is the immersed depth of the capillary #1,  $\Delta h$  is the difference of immersed depth of the capillaries #1 and #2,  $r$  and  $r'$  are the inner diameters of the capillaries #1 and #3.



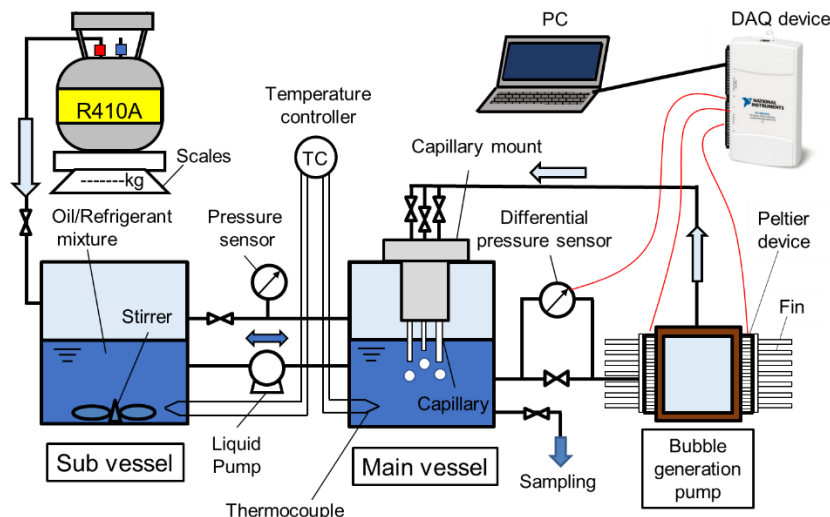
**Figure 2:** 3-capillary method

### 2.3 Experimental setup

Figure 3 shows an experimental apparatus used to measure the surface tension of oil/refrigerant mixture in this study. The experimental apparatus mainly consisted of a refrigerant cylinder, a sub vessel, a main vessel, a differential

pressure transducer and a bubble generation pump. Capillary tubes of 0.28 mm and 0.45 mm in inner diameter were set in the main vessel. Refrigeration oil was polyvinyl ether (PVE) and refrigerant was R410A. The sub vessel was used to prepare the PVE/R410A mixture. After storing a given amount of PVE oil in the sub vessel, R410A was supplied from the refrigerant cylinder to the sub vessel. R410A was dissolved into the PVE using a magnet stirrer. After the PVE/R410A mixture reached equilibrium condition under the given pressure and temperature in the sub vessel, the mixture was sent to the main vessel via a gear type oil pump, and the oil level in the main vessel was adjusted so that the tips of the capillary tube were immersed in the oil/refrigerant mixture. The immersed depth of the capillary #1 in the mixture was set to be about 10 mm and was measured by a CCD camera through a sight glass mounted on the main vessel. The accuracy of measurement of the immersed depth was 0.03 mm. The upper part of the main vessel was connected to a bubble generation pump described in the next section, and the pump was connected to the capillary tubes to generate the bubble at the capillary tubes. The pressure difference between the main vessel and the capillary tube was measured by the pressure differential transducer. The accuracy of the pressure differential sensor was within 0.25% of full scale. A tape heater and an insulating urethane foam were wrapped around the main vessel, and the temperature of the test section was kept constant by a digital temperature controller. Lines outside the main vessel and the pressure differential transducer were also heated to prevent the condensation of the refrigerant at the lines and the transducer. The temperature was measured by a T-type thermocouple and the measurement error of the thermocouple was within 1.0 °C. The pressure was monitored by a strain-gauge type pressure gauge with an accuracy of 0.25% of full scale.

The pressure inside the capillary tube becomes maximum one when the bubble radius becomes equal to the inner radius of the capillary during the bubble generation cycle. By measuring the maximum bubble pressure, the surface tension of the mixture was calculated from Equation (4) with the density of the mixture and the immersed depth of the capillary obtained by Equations (2) and (3). The concentration of refrigerant in the mixture was measured by a weighing method and checked by a sampling method. In the experiment, the measurements of the surface tension and the refrigerant concentration of the mixture were carried out in the pressure range up to 3.5 MPa and temperature range from 30 to 90 °C. Viscosity grade of the PVE used in this study was  $68 \text{ mm}^2\text{s}^{-1}$  at 40 °C. The pressure difference and the temperatures were acquired by a PC through a DAQ device.



**Figure 3:** Experimental apparatus of surface tension measurement

## 2.4 Bubble generation pump

In the maximum bubble pressure method, the bubble generation pump is necessary to generate the bubble at the capillary tube. It should work under high pressure and temperature condition to apply the method to the oil/refrigerant mixture, and without pressure pulsation for stable measurement. In addition, small size, simple structure and easy pressure control are desirable. In this study, the bubble generation pump using Peltier elements was developed. Figure 4 shows a schematic diagram of the pump. Two Peltier elements and fins were attached by adhesive on side hollows of a copper container in which the refrigerant gas was filled. The Peltier elements heated the copper container with applying electric current to the elements, and the temperature inside the container increased. When the Peltier elements

were turned on with closing a valve between the pump and the main vessel, the pressure inside the container increased and the refrigerant gas was fed to the capillaries to generate the bubble. The electric current to the Peltier element was controlled by a solid-state relay with a pulse width modulation (PWM) signal from the PC. Figure 5 shows a time variation of the pressure inside the pump for different duty ratio of the PWM signal when the Peltier element was turned on from the equilibrium condition at the room temperature. It can be seen that the pressure inside the pump was controllable with changing the duty ratio of the PWM signal and the bubble generation interval at the capillary tube could be controlled by the pressure in the pump.

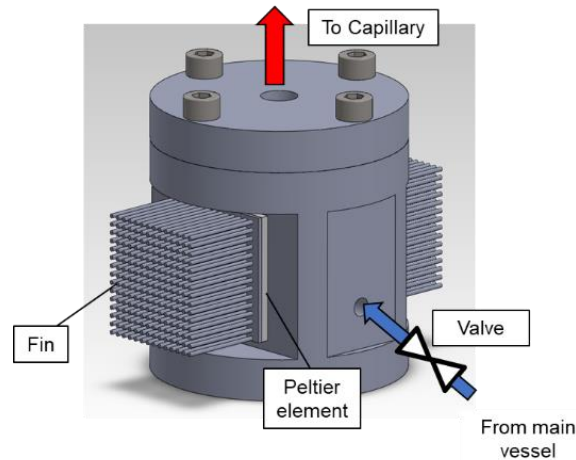


Figure 4: Bubble generation pump with Peltier elements

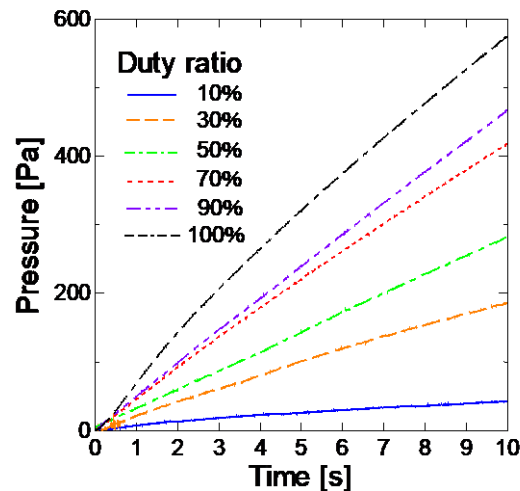


Figure 5: Pressure change inside bubble generation pump

### 3. RESULTS AND DISCUSSION

#### 3.1 Measurement of density

In the calculation of maximum bubble pressure method, the density of oil/refrigerant mixture is required. The density of oil/refrigerant mixture was calculated by the following equation from the refrigerant concentration and both densities of PVE and R410A.

$$\rho = \frac{M_{ref_1} + M_{oil}}{k \frac{M_{ref_1}}{\rho_{ref_1}} + \frac{M_{oil}}{\rho_{oil}}} = \frac{\rho_{ref_1} \rho_{oil}}{\rho_{ref_1} (1-C) + k \rho_{oil} C} \quad (5)$$

where,  $M_{ref_1}$  is the mass of refrigerant dissolved in the oil,  $\rho_{ref_1}$  is the density of liquid refrigerant.  $M_{oil}$  and  $\rho_{oil}$  are mass and density of the oil, respectively. It is well-known that the volume of oil/refrigerant mixture is less than the simple sum of each volume of oil and liquid refrigerant. Coefficient  $k$  in Equation (5) indicates the influence of volume reduction and it was found to be 0.97 by a preliminary test in which the volume increase of oil was compared with the volume of liquid refrigerant based on the concentration obtained by the sampling method.

Figure 6 shows the saturated liquid density of R410A calculated by REFPROP (Lemmon, *et al.*, 2013). Since the critical temperature of R410A is 71.8°C, the density above this temperature cannot be defined. Fukuta *et al.* (2004) reported that since refractive index of the PVE/R410A mixture showed continuous variation against the temperature even at the super-critical temperature, it was supposed that the dissolved refrigerant exists in the oil in a condition like liquid refrigerant even at the super-critical temperature and the refractive index of the refrigerant at the super-critical temperature, therefore, could be calculated using a linear extrapolation of the refractive index of the saturated liquid refrigerant in a subcritical temperature range. In this study, the density of R410A above the critical temperature was estimated by extrapolating linearly from the density below the critical temperature (0-50°C). The estimated density is shown by a broken line in Figure 6. From the densities of PVE and R410A, the density of the PVE/R410A mixture was calculated from Equation (5). The density of the mixture was measured by applying the MBPM with the capillaries of different immersed depth as described by Equation (2).

Figure 7 shows the density of PVE/R410A mixture at 30 and 90°C. The line shows the estimated density at each temperature in the manner above mentioned. The measured density agrees with the estimated one and it was found that the density of the mixture can be calculated by Equation (5) even at the temperature above the critical temperature using the linear extrapolation of the density of the saturated liquid refrigerant at the subcritical temperature. The density of mixture changes linearly with the refrigerant concentration and the tendency against the concentration depends on the temperature. Although the measured density scattered especially in 90°C, the influence of error of the density on the calculation of surface tension in Equation (4) is very small, *e.g.*, the density error of 3% causes the surface tension error of 0.05%. Since the variation of the density of the mixture due to the refrigerant dissolution is not so large and the influence of the density on the surface tension is very small, a constant density can be used in the calculation of the surface tension if the density of the mixture is unknown.

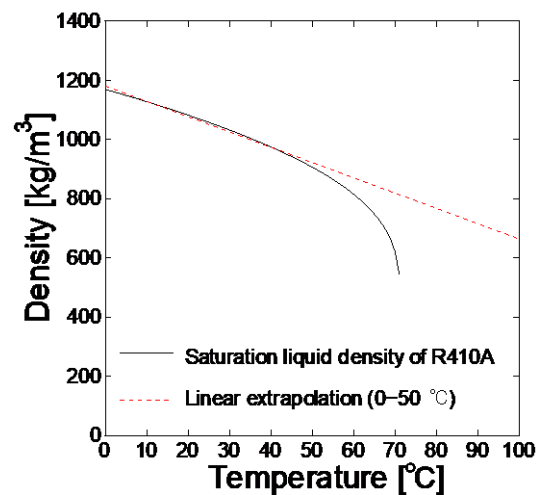
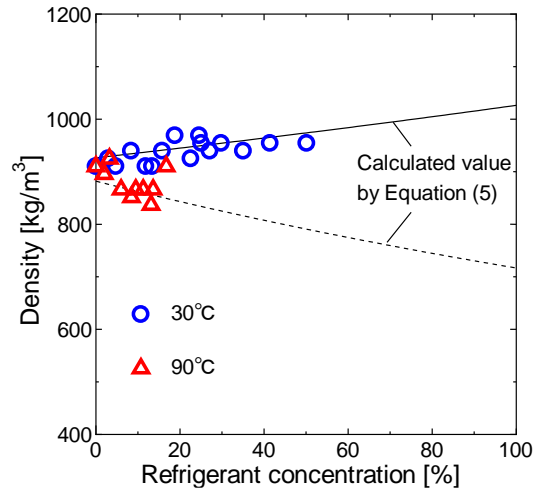


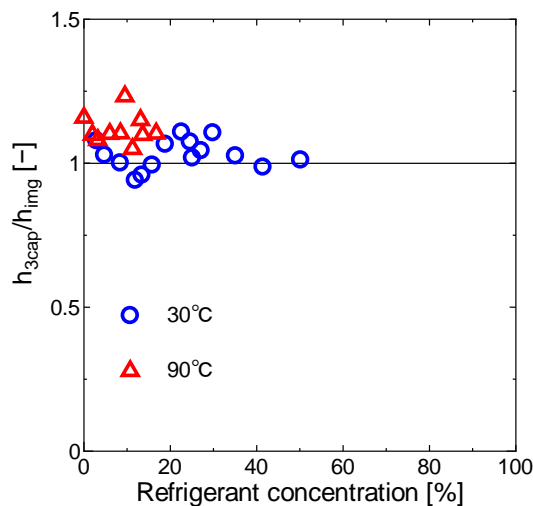
Figure 6: Relationship between refrigerant density and temperature



**Figure 7:** Density of PVE/R410A mixture

### 3.2 Measurement of oil level

The immersed depth of the capillary tube is calculated from Equation (3). Figure 8 shows the immersed depth of capillary tube versus the refrigerant concentration at 30 and 90°C. The immersed depth of capillary in Figure 8 is normalized by that observed from the sight glass. The maximum error in measuring the immersed depth of capillary tube by the 3-capillary method was 20%, and the average error was about 10%. When the difference of inner diameters between the two capillary tubes increases, the accuracy of immersed depth measurement is improved. However, the accuracy of surface tension measurement decreases by a capillary with larger diameter, while flow resistance increases and clogging tends to occur when the inner diameter of capillary is too small. Although 10% error in the immersed depth measurement is not so good, the oil level above the capillary tip in the compressor shell can be approximately measured when the capillary tubes with different diameter are mounted inside the compressor shell. The 10% error of the immersed depth measurement of capillary by the 3-capillary method results in 3% error of the surface tension measurement and 2% error in a measurement of refrigerant concentration in the oil/refrigerant mixture.



**Figure 8:** Capillary depth of PVE/R410A measured by 3-capillary method

### 3.3 Measurement of surface tension

Before measuring the surface tension of PVE/R410A mixture, the surface tension of PVE was measured. Figure 9 shows the surface tension of PVE along with that of the saturated liquid of R410A obtained by REFPROP. The surface tension of PVE decreases linearly with temperature. The surface tension of R410A is smaller compared to that of oil, and becomes  $0 \text{ N m}^{-1}$  at the critical temperature. The surface tension of refrigerant in the region above the critical temperature cannot be defined and was regarded as  $0 \text{ N m}^{-1}$  in this study.



Figure 10 shows the surface tension of PVE/R410A mixture against the refrigerant concentration measured at 30 - 90°C. The condition of the mixture was under the saturated condition. The surface tension of refrigerant is much smaller than that of oil as shown in Figure 9, and the surface tension of PVE/R410A mixture decreases steeply as the refrigerant concentration in oil increases. Since the surface tensions of oil and refrigerant decreases with temperature, the surface tension of the mixture also decreases with temperature at the same refrigerant concentration. The change of surface tension due to the mixing of refrigerant was normalized by the difference of surface tensions of oil and refrigerant at each temperature, and the normalized surface tension is plotted against the refrigerant concentration in Figure 11. The relationship between the surface tension of the mixture and the refrigerant concentration was correlated by the following equation proposed in the previous study (Fukuta *et al.*, 2017).

$$\frac{\gamma - \gamma_{ref}}{\gamma_{oil} - \gamma_{ref}} = (1 - C)^n \quad (6)$$

where,  $\gamma$ ,  $\gamma_{ref}$  and  $\gamma_{oil}$  are surface tensions of the mixture, refrigerant, and oil, respectively.  $C$  is the refrigerant concentration in the mixture and  $n$  is an index factor. The index factor  $n$  was determined as 3.33 for the combination of PVE and R410A by the least square method with the measured surface tensions. The normalized surface tension calculated by Equation (6) using the index factor of 3.33 is shown in Figure 11 as well as those using the indices of 2 and 4 as a reference. It was found that the normalized surface tension was well correlated by Equation (6) with the index factor of 3.33 at all temperatures.

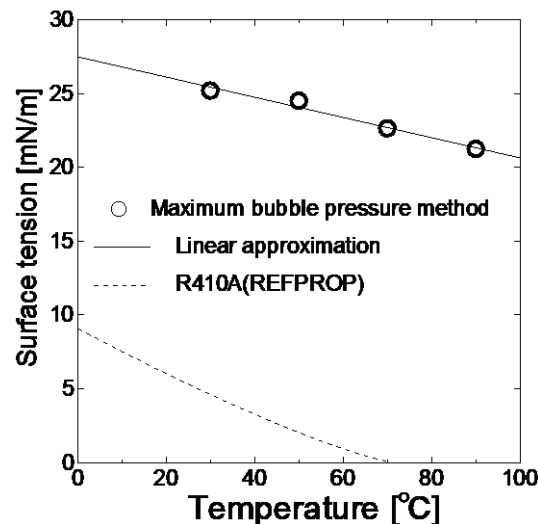
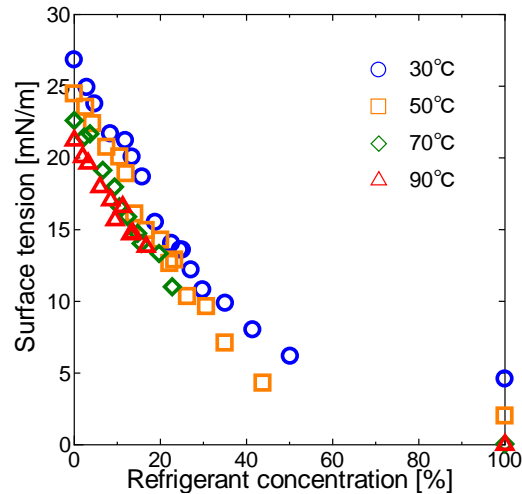
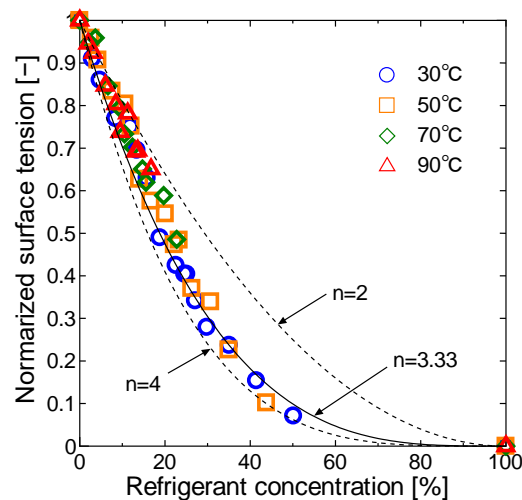


Figure 9: Result of maximum bubble pressure measurement



**Figure 10:** Result of surface tension measurement



**Figure 11:** Correlation between refrigerant concentration and surface tension

#### 4. CONCLUSIONS

Surface tension of PVE/R410A mixture was measured by the maximum bubble pressure method (MBPM) and the 3-capillary method was applied to measure the density of the mixture, the immerse depth of the capillary and the surface tension of the mixture simultaneously. The following conclusions were obtained.

1. A Peltier pump for bubble generation in the MBPM was developed, in which a copper container of the pump was heated by Peltier elements, temperature of refrigerant gas in the pump increased and pressure inside the pump increased by thermal expansion of the refrigerant gas. The bubble generation interval at a capillary tube could be controlled by an electric current supplied to the Peltier element.
2. Density of PVE/R410A mixture was calculated and compared with the experimental results. It was found that the density of mixture can be calculated by Equation (5) even at the temperature above the critical temperature using the linear extrapolation of the density of the saturated liquid refrigerant at the subcritical temperature.
3. Oil level of PVE/R410A mixture was obtained by Equation (3) in the 3-capillary method and compared with observed oil level. It was found that the 3-capillary method can measure an approximate oil level in a compressor shell. The influence of the immersed depth of capillary on the surface tension measurement is small.

4. The surface tension of PVE/R410A mixture was measured from 30 to 90°C. The surface tension of PVE/R410A mixture decreased steeply as the refrigerant concentration in oil increased. The change of surface tension by mixing the refrigerant was normalized by the difference of surface tensions of oil and refrigerant, and the normalized surface tension was successfully correlated with the concentration of refrigerant in oil by Equation (6) with an index factor of 3.33 in a temperature range from 30 to 90°C.

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