A dynamic optical measurement system for cryogenic fluids using laser interferometry

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Abstract. Dynamic visualization is of great significance in the research of flow conditions and mass transfer process of cryogenic fluids. In this paper, two common ways to measure the concentration of cryogenic fluids are introduced and compared. To improve the real-time monitoring of cryogenic fluid, a non-contact dynamic optical measurement system using laser interferometry is designed, which is sensitive to subtle changes of fluid concentration. A precise and dynamic interference pattern can be obtained using this system. Two-dimensional concentration distribution of the fluid can be calculated from the interference pattern. Detailed calculation process is presented in the paper.

1. Introduction
The Cryogenic liquids (such as liquid oxygen, liquid nitrogen, etc.) are widely used in steel industry, chemical industry, food industry and aerospace defense industry, etc. Dynamic monitoring is of great significance in the research of flow condition and mass transfer process of cryogenic fluids. However, due to the problems of sealing and thermal insulation, it's lack of an effective real-time monitoring method for cryogenic fluid behaviors, especially for subtle changes. In this paper, we designed an innovative measurement system based on laser interferometry to solve the problem.

2. Cryogenic fluid concentration measurement methods
At present, sampling and capacitance method are common ways to measure the concentration of cryogenic fluids.

Gas chromatograph (GC) has been used to analyse the sample for a long time and it is widely applied to measure the impurity concentration of liquid air separation products [1-2]. When the sample goes through the gas chromatograph under the drive of the carrier gas, components will get different corresponding speeds according to their interaction with the packing of chromatographic column, and the detector will detect out the component concentration. The accuracy commonly reaches ppm level for sampling method, but the sampling devices will disturb the fluid and can’t provide dynamic information.

Capacitance-type densimeters using changes in capacitance, such as those for the measurement of two-phase hydrogen density [3] and LNG density [4], generally employ either a double cylinder or a
parallel plate structure [5]. Capacitance has a linear relation with specific dielectric constant which is unique for a specific substance. Therefore, capacitance varies when different concentration fluid flows through the electrodes. Capacitance method can provide real-time concentration change of fluid and could get an accuracy of $10^{-4}$ [6], but the measured concentration is average value between the electrodes, so it can’t provide the distribution of fluid concentration.

Laser interferometry utilizes refractive index to measure liquid concentration, usually at room temperature [7-8]. Interferograms of liquid air had been obtained in 2004 [9], that proved laser interferometry could be utilized for the measurement of cryogenic fluid. Refractive index is an important parameter. Under certain condition, the change of concentration could be derived from the change in refractive index. The theoretical calculation in solution refractive index has been pretty mature, and Lorentz-Lorenz formula is applied extensively in the calculation of multicomponent liquid refractive index for now, whose absolute deviation is only about $10^{-4}$ [10-11]. Based on this, a non-contact dynamic optical measurement system was designed. Interference pattern can be obtained when a laser beam through the cryogenic liquid meets its coherent light. The dynamic changes of the interference pattern depend on the changes of the optical path difference, which are caused by the changes of cryogenic liquid refractive index. So we can get the dynamic subtle changes of cryogenic liquid components. Detail comparison is shown as table 1.

Table 1. Comparison of different concentration measurement methods

| Concentration measurement methods | Advantages                  | Disadvantages                              | Accuracy |
|----------------------------------|-----------------------------|--------------------------------------------|----------|
| Sampling with GC                 | Convenient, high precision  | Only for spot information, discontinuous   | PPM−PPB  |
| Capacitance method               | Simple installation, high   | Can’t provide concentration distribution   | $10^{-4}$|
|                                  | real-time monitoring        |                                            |          |
| Laser interferometry             | Real-time monitoring, high  | Complex data processing, rarely applied to | $10^{-4}$|
|                                  | concentration detection     | cryogenic fluid                           |          |

3. Experimental setup
This measurement system mainly includes two parts: cryogenic system and optical system.

3.1. Cryogenic system
The cryogenic system mainly consists of three parts: cryogenic solution preparation, test chamber and data acquisition, as shown in figure 1. Liquid oxygen and nitrogen are pumped into a dewar, respectively. The dewar is put on a wide-range electronic scale to prepare specific concentration mixture. As the mixture flows over the magnetizer, liquid oxygen will be attracted because of its paramagnetism, which leads to a subtle change of mixture concentration. There are four parallel windows placed in the test chamber shown as figure 2 that allow the laser to go through. Glass-ceramic welded with Kovar is used for inner windows to ensure the sealed capability at liquid nitrogen temperature. Outer windows are made of high transmittance optical quartz glasses sealed with PTFE gasket and fluorine rubber O-ring on each side. The diameter of inner window is 35 mm and the distance between the inner windows is 12.7 mm. The magnet field generated by NdFeB is imported into the chamber by 79 permalloy. Gas chromatograph is used to measure the mixture concentration in dewar and test chamber, intermittently, to verify the result of laser interferometry. Data of flow rate, temperature and pressure are collected by Keithley 2700/7700 data acquisition system.
3.2. Optical system

The optical system shown in figure 3 is based on Mach-Zehnder interferometer. The laser passes through the beam expander and then is divided into two beams by the first beam splitter. One beam goes through the test chamber, carrying the liquid concentration information, meets the other coherent light from the second beam splitter, and produces interference pattern on the screen. As the laser goes through the fluid being tested just once (twice in many other interferometers [12-13]), Mach-Zehnder interferometer is insensitive to external disturbances, which is well suited to measure cryogenic fluid which can be easily disturbed by the heat leakage from atmosphere.

A 532 nm semiconductor laser was chosen to avoid the absorption peaks of liquid oxygen. The laser unit—with the power of 10 mW could generate linear polarization laser (>100:1) to get fairly...
good spatial coherence. To get clear and steadily interference pattern at the second beam splitter, a n:1 \((n > 1)\) first beam splitter could be used to make more light go through the test chamber to offset the dissipation in test chamber. The four mirrors must be strict 45 degrees so that the two light beams can overlap completely at the screen.

**Figure 3.** Optical path diagram

4. Theoretical calculation and discussion

4.1. Theoretical calculation

One of the most frequently used mixing rules in the analysis of refractive index and density data of mixtures is Lorentz-Lorenz Rule

\[
\frac{n^2 - 1}{n^2 + 2} = \sum_{i=1}^{N} \Phi_i \frac{n_i^2 - 1}{n_i^2 + 2}
\]  

(1)

Here, \(n, n_i\) are the refractive indices of the mixture and component \(i\), \(\Phi_i\) is the volume fraction of the component \(i\). As for our measurement system, Lorentz-Lorenz formula changes to

\[
\frac{n^2 - 1}{n^2 + 2} = x_1 \frac{n_1^2 - 1}{n_1^2 + 2} + x_2 \frac{n_2^2 - 1}{n_2^2 + 2}
\]

(2)

Here, \(n, n_1, n_2\) are the refractive of mixture and liquid oxygen and liquid nitrogen, \(x_1, x_2\) are the volume fractions of respective components. So we can get

\[
n^2 = x_1 n_1^2 + x_2 n_2^2
\]

(3)

\[
n = [x_1 (n_1^2 - n_2^2) + n_2^2]^{\frac{1}{2}}
\]

(4)

\[
n' = \frac{1}{2} [x_1 (n_1^2 - n_2^2) + n_2^2]^{-1/2} (n_1^2 - n_2^2)
\]

(5)

The corresponding change of an interference fringe spacing

\[
\Delta n \cdot d = \lambda
\]

(6)

\[
\Delta x_1 \cdot n' = \lambda / d
\]

(7)

Here, \(d\) is the liquid thickness, \(\lambda\) is the wave length of laser, \(\Delta x_1\) is the volume fraction change of \(x_1\)

\[
\Delta x_1 = \frac{2\lambda}{d(n_1^2 - n_2^2)} [x_1 (n_1^2 - n_2^2) + n_2^2]^{\frac{1}{2}}
\]

(8)
Since the $\lambda$, $d$, $n_1$, $n_2$ are all known and $x_1$ can be measured by GC (as shown by figure 1), we can calculate the $\Delta x_1$.

4.2. Discussion

Laser interferometry is sensitive to subtle changes, will not disturb the cryogenic fluid and can provide two-dimensional real-time information for large areas. Different from common fluids at room temperature, cryogenic fluids can be easily disturbed by subtle heat leakage from atmosphere, which can lead to confusion of interference pattern. So the performance of heat insulation is extremely important for the test chamber and the vacuum degree should reach to $10^{-3}$ Pa level at least. The inner window is a key point which needs a special attention because it’s where cryogenic fluid, glass and 304 steel act upon each other.

Although the interference pattern captured around the magnetizer can show the variation tendency of concentration, the accurate change value is desired. For now, the accuracy is limited by refractive index, as there are not enough data at low temperatures. We only get that the refractive index of nitrogen is 1.19876 at 77 K, 578 nm, and of oxygen is 1.2243 at 90 K, 546 nm [14]. Since the 532 nm laser was used at mixture temperature, the effect of wavelength and temperature on refractive index was ignored passively. In the next step, series of experiments will be operated and the concentration data around the magnetizer measured by gas chromatograph will be used to compare with the result deduced by the interference pattern under the same condition. Then a correction of Lorentz-Lorenz formula applied to cryogenic fluid could be made.

5. Conclusion

In summary, a novel non-contact dynamic optical measurement system is constructed to measure the subtle change of cryogenic fluid concentration, and it’s flexible to study the influence of gradient magnetic field on paramagnetic fluids. However, to increase the precision of measurement, more work is needed to find a proper correlation of refractive index and concentration in cryogenic fluid.

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