Research on a Method for the Determination of Iron in Lubricating Oil

Liao Qiang
College of Mechanical Engineering & The State Key Laboratory of Mechanical Transmission
Chongqing University
Chongqing P.R. China 400044
lq922@163.com

Li Xiaowei
College of Mechanical Engineering
Chongqing University
Chongqing P.R China 400044
Lee772889829@126.com

Chen Lili
College of Mechanical Engineering
Chongqing University
Chongqing P.R China 400044

Wu Jiang
College of Mechanical Engineering
Chongqing University
Chongqing P.R China 400044

***Abstract*—Mechanical impurity and iron in particular of lubricating oil will increase, if transmission system in mechanical devices is worn. Therefore, it is particularly important to measure iron content of lubricating oil. There are several methods for detecting lubricating oil at present, but these methods have some disadvantages such as high cost, complex operation, long testing cycle and so on. The paper puts forward the impedance method for measuring iron content of lubricating oil. Based on a series of experiments of the prepared sample oil of different iron content, the paper discussed the excitation current, suitable measurement frequency, and analyzed correlation between iron content and impedance, thus establishing mathematical model between iron content and impedance. The experimental results showed that it is feasible to use the impedance method for determination of iron content in lubricating oil.**

Keywords- Impedance method; iron content; lubricating oil; mechanical devices

I. INTRODUCTION

Lubricating oil is to machine what blood is to man. It plays an important role in sealing, lubrication, cooling, cleaning, shock-absorbing and anticorrosive in the most mechanical devices[1].There are some indicators to evaluate lubricating oil, such as moisture content, total acid value, mechanical impurity and so on[2]. Much metal grit can be found because of the friction of parts and other mechanical impurities when equipments work for a long time [3]. Deposit sediment in oil will increase dramatically if the bad oil continues to be used, thus machine parts are worn and even the machines don't work [4]. In order to ensure safety and stable operation of machine, lubricating oil should be renewed timely. The national standard GB/T7607-2002 makes detailed index renew lubricating oil, and iron is an important indicator. Therefore, it is of great significance to measure iron in lubricating oil.

It is very important to check pollution of lubricating oil, and there are mainly have the following several methods for detecting lubricating oil at present [1].

1) The method of physical and chemical analysis. Physical and chemical analysis is the method analyzing viscosity, flash point, moisture, total acid value, metal abrasive and other indicators of sample oil through instrument in the laboratory.

2) Spectral analysis methods. It includes atomic emission spectrometry, atomic absorption spectrometry, infrared spectrum analysis and X-ray fluorescence spectrometry.

3) Online iron spectral instrument. Ferrous spectrum technology separate metal grit from lubricating oil and arrange according to the size by magnetic gradient and gravity gradient.

4) Monitoring of electrical method. It mainly includes resistive method and capacitive method.

The methods introduced above have some disadvantages such as high cost, complex operation, long testing cycle and so on. The paper researched a method for determination impedance of lubricating oil so as to measure iron content and determine whether to change oil. The new oil conductive performance is poor, namely its impedance is high. The impedance will change when the sample oil is added iron powder, thus the impedance can be used to analyze iron content of sample oil.

II. EXPERIMENT AND METHOD

A. The main experimental equipment

The Agilent 4294A precision impedance analyzer,16452 liquid test fixture, computer, small beaker of 10ml, pipette, electronic analytical balance, glass rod and so on.

B. Experimental material

Lubricating oil of Shell 5W-30, Black iron powder that the iron content less than 98% (Contains very small amounts of copper, soluble in water and other impurities).

C. Sample preparation

The experiment consisted of 7 samples with different iron content, and the iron content is 0, 0.025%, 0.15%, 0.5%, 0.75%, 1%, 2%.

D. *The process of determination*

Add the prepared sample oil to the liquid fixture and measure the impedance of lubricating oil by Agilent. Then measure the impedance of lubricating oil in the conditions of different excitation current and different frequency in the process of whole experiment.

Firstly, set the excitation current, which ranged from 200uA to 20mA. The relationship between impedance and current would be analyzed to determine excitation current through measuring impedance in different frequency. The next, make frequency band ranges from 40 Hz to10 kHz at the determined excitation current. It was worth putting forward that each of sample oil should be measured 5 times fast because of the deposition phenomenon of iron [1]. In fact, it is not difficult to think that there will be downward trend because of the properties of iron. However, this phenomenon can be affected by the viscosity of lubricating oil, the iron particle size and time. Considering the experimental oil and diffusion capacity or adhesion ability of iron powder [5], the average value of 5 times should be calculated to build mathematical model between impedance and iron content. Thirdly, proper frequency would be determined through observing the regularity between impedance and frequency and the correlation coefficient of impedance and iron content. Then, establish mathematical model between the impedance and iron content in the condition of the determined excitation current and suitable measurement frequency. Lastly, analyze error of determination of iron in lubricating oil.

III. RESULTS AND DISCUSSION

A. *The impact of the excitation current*

The frequency of 1 kHz and 10 kHz were selected to analyze the relationship between impedance and current. The result was given as Figure 1 and Figure 2, which can represent regularity of all frequencies.

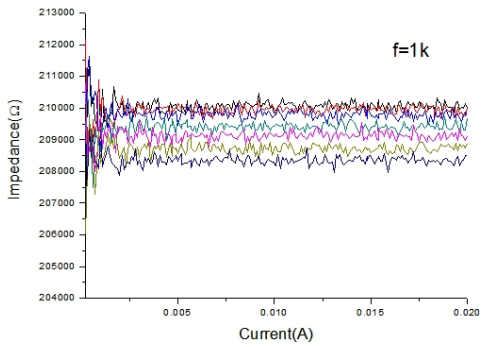

Figure 1 The relationship between impedance and current

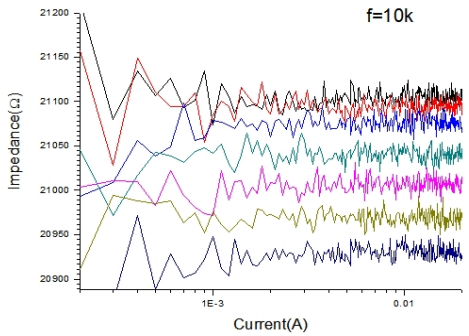

Figure 2 The relationship between impedance and current

Each of sample oil was chosen to describe the relationship between the impedance and current in different frequency. Take sample oil of iron content of 2% as example and the result was given as Figure 3. It can represent regularity of all sample oils.

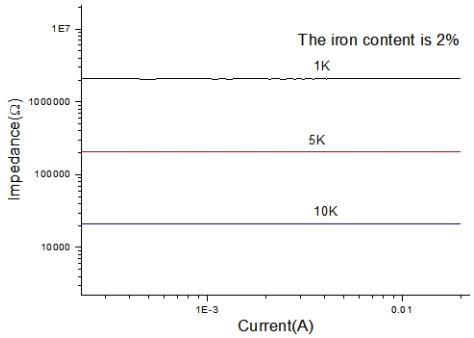

Figure 3 Impedance changes with the current in different frequency

The figure 1 and 2 show that impedance almost have no change when the current was above 1mA.The figure 3 show that impedance would change when frequency was low. Therefore, the experiment should be done in higher current so as to decrease the impact of the excitation current. Moreover, high frequency should be selected and the current was set up for 20mA, which is the largest value the instrument can provide.

B. *Suitable measurement frequency*

The impedance of lubricating oil changes with the variation of iron content in different frequency. The measurement frequency band was divided into high and low frequency band and the critical point was 2 kHz. The relationship between impedance and frequency was given as Figure 4 and Figure 5.

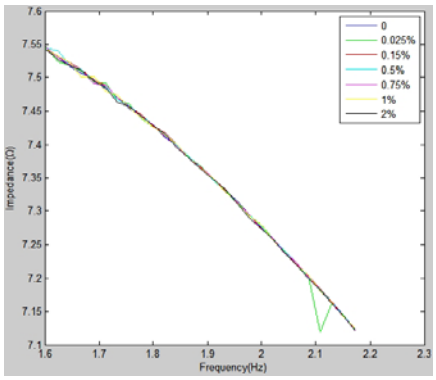

Figure 4 Impedance changes with low frequency band

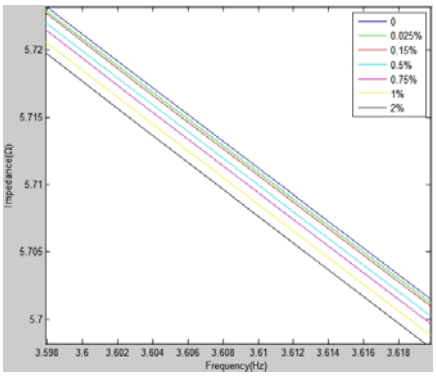

Figure 5 Impedance changes with high frequency band

The figure 4 show that the impedance changes irregularly in low frequency band. On the contrary, the figure 5 shows that the high frequency band is suitable measurement frequency and indicates that impedance became smaller with the increase of frequency for each kind of sample oil. Moreover, in order to obtain the total information of the frequency band, the correlation between iron content and impedance should be analyzed. 20 points were analyzed by SPSS 19.0. The result was given as table1.

TABLE1. CORRELATION COEFFICIENT BETWEEN IRON CONTENT AND IMPEDANCE

Frequency (Hz)	Correlation coefficient	Frequency (Hz)	Correlation coefficient
54.20	-0.357	2542.23	-0.948
81.27	-0.089	2959.27	-0.962
121.85	-0.567	3444.72	-0.930
182.71	-0.530	4009.80	-0.964
260.42	-0.904	4910.00	-0.955
390.48	-0.848	6012.29	-0.947
556.58	-0.940	6998.57	-0.954
793.33	-0.947	8146.64	-0.946
1251.30	-0.962	9014.84	-0.965
1973.64	-0.939	9975.56	-0.958

The table 1 showed that the frequency band ranging from 2k to 10k had the better correlation, that is to say, the frequency ranging from 2 kHz to 10 kHz was suitable measurement frequency.

In summary, the current was set up for 20mA, and the frequency ranged from 2 kHz to 10 kHz. It is feasible to use the impedance method for measuring iron content of lubricating oil.

C. *The relationship between impedance and iron content*

The data show that correlation between impedance and different iron content are closed as a whole. So any frequency can be selected to describe the relationship between impedance and iron content. Therefore, one point was chosen to fit curve equation of two variables [6]. The fitting curve was given as Figure 6(Take f=4910.0 Hz as example).

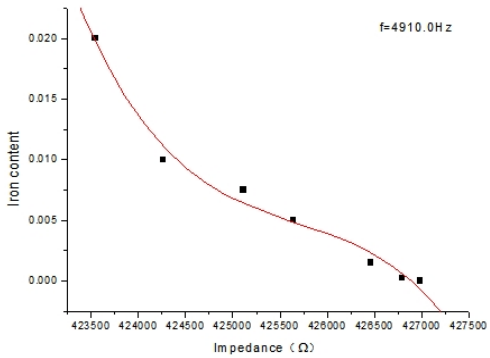

Figure 6 Fitting curve (f=4910.0 Hz)

The fitting equation was given below (where Y and X represent iron content and impedance). The equation would be used to calculate the iron content if know impedance in a certain range of iron content.

$$Y = 75184.6 - 0.6X + 1.2\times10^{-6}X^2 - 9.7\times10^{-13}X^3$$

D. *Error analysis of determination of iron content of lubricating oil*

There were some factors led to uncertain errors for the determination of iron content of lubricating oil, such as the weighting error the sample, the purity of standard storage solution, the fitting standard curve and so on, which also have a lot of reference for study on the impedance method for determination of iron content of lubricating oil [7]. These factors really led to some data points irregular. In addition, the impedance of different iron in sample oil would also be affected if the sample oil was inhomogeneous mixture and generated bubble phenomenon in the process of experiments. Despite of these factors, general trend of the experimental data was correct, which showed the impedance method for measuring iron content in lubricating oil is feasible.

IV. CONCLUSIONS

According to the analysis of experimental result, the condition of applying the impedance method for determination of iron in lubricating oil was that the excitation current was 20mA and suitable measurement frequency ranged from 2 kHz to 10 kHz. The impedance and iron content had good relevance and the average correlation coefficient was -0.953 in that condition. The impedance method can measure iron content of lubricating oil in a certain scope by fitting the curve equation between iron content and impedance, and the curve showed the impedance of sample oil became small with the increasing of iron content. However, the relationship between iron content and measurement error was inverse proportion by using the impedance method for the determination of iron content, thus we need to research further so as to extend the measurement range.

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