Design of a Hydrogen Content on-line Measurement System in the Process of Steel Heat Treatment

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***Abstract*-This paper describes a hydrogen content on-line measurement system in the process of steel heat treatment based on the theory of hydrogen concentration cell. This System design includes two main measurement units and a MCU for calculation and output. The method of measurement data processing is discussed in this paper, and an algorithm of temperature value calibration is also proposed. Final test shows that the instrument has features of portability, continuity and real time capability, which make it has a potential future in the hydrogen content measurement in metallurgical enterprises.**

Keywords-Hydrogen content measurement, Hydrogen concentration cell, Portable instrum-ent, Temperature calibration, Piecewise function approximation algorithm

I. INTRODUCTION

Because of the low solubility of hydrogen in solid steel, it would be precipitated in the process of solidification and cooling of molten steel with some gas such as CO, N_2 etc [1]. The precipitated hydrogen would cause some defects such as subsurface blowholes、central pipe and porosity, and these defects would lower the intensity and plasticity of the steel, which is so called hydrogen embrittlement phenomenon [2]. It is very helpful to design an instrument which can measure the hydrogen content in the process of steel heat treatment. However, traditional equipments in this field such as SLM and NOTORP [3,4] are normally too cumbersome and expensive, in this paper a portable and low cost design for the measurement of hydrogen is proposed.

II. OPERATING PRINCIPLE

Hydrogen sensor is designed based on the theory of hydrogen concentration cell, as Figure.1 shows. The shadow part is the proton conductor electrolyte. The high hydrogen differential pressure side ($P^{\mathrm{I}}_{\mathrm{H_2}}$) is anode and the low hydrogen differential pressure side ($P^{\mathrm{II}}_{\mathrm{H_2}}$) is cathode, so the cell reaction formula is given:

$$H_2(I)=H_2(II) \quad (1)$$

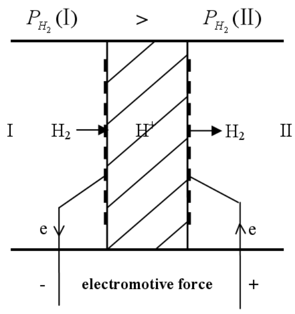

Figure 1. Illustrative diagram of hydrogen concentration cell

Because of the difference of hydrogen chemical potential between the two eletrodes, there would be generated different electrode potential caused by the electrochemistry reactions at the boundary between both of electrodes and the electrolyte. According to Nernst formula:

$$E = \frac{RT}{2F} ln \frac{P^{\mathrm{I}}_{\mathrm{H_2}}}{P^{\mathrm{II}}_{\mathrm{H_2}}} \quad (2)$$

where E is the electromotive force of the hydrogen concentration cell, R is the universal gas constant ($8.314 J\cdot mol^{-1}\cdot K^{-1}$), F is the faraday's constant ($96500 C\cdot mol^{-1}$) and T is the absolute temperature. $P^{\mathrm{I}}_{\mathrm{H_2}}$ and $P^{\mathrm{II}}_{\mathrm{H_2}}$ (*atm*) are the hydrogen partial pressure of either electrode, which one is the certain reference electrode, the other is measurement electrode. So as long as the E and T are measured, the hydrogen partial pressure of measurement electrode will be calculated by formula (2), and the final measurement results will be presented in logarithm form according to custom.

III. SYSTEM DESIGN

As mentioned above, the main objective of the system is measuring the two parameters: E and T. The system chart of the design is shown in Figure.2.

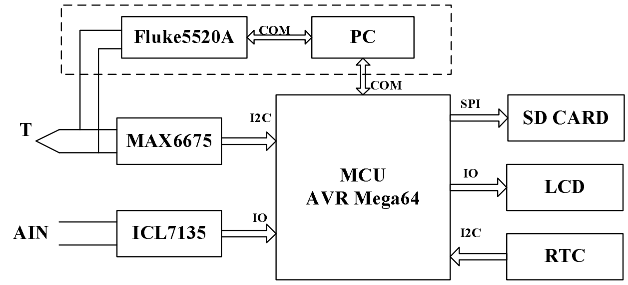

Figure 2. The chart of the system

This MCU-based system consists of several modules, of which the electromotive force measurement module and the temperature measurement module are in charge of measuring the two expected parameters, the LCD module and SD card module are in charge of showing and storing the calculated results respectively. There are also other modules such as real-time clock module, key input module and power supply module and so on. Most of the units are connected with a micro controller unit, ATmega64, which is a high-performance, low-power Atmel AVR 8-bit microcontroller with 64K bytes in-system programmable flash. The unit inside the dotted box as Figure.2 shows only operates in the calibration phase. The calibration phase works before the real measurement phase. When the system is powered up, it reads the key input to switch between the two phases. The main measurement program of the microcontroller consists of reading all the input measurement parameters and calculating, then showing the final results on the LCD and storing the data into the SD card, as Figure.3 shows. The calibration program will be discussed later.

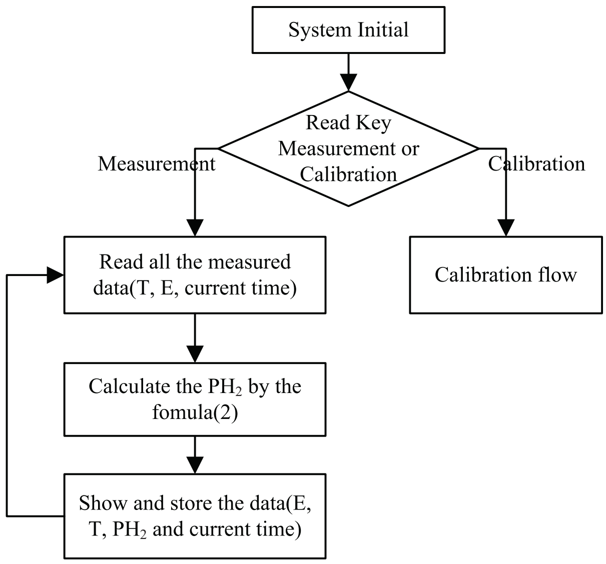

Figure 3. The flow chart of the system main program

In all of above mentioned units, the two measurement units are the most important. These units will be discussed in detail respectively.

A. electromotive force measurement unit

The main job of electromotive force measurement unit is measuring a variable DC voltage input, because the resistance of the hydrogen concentration cell is high, in order to obtain accurate eletromotive force data, the measuring circuit needs very high input resistance. So a precision AD converter with $4^1/_2$ digit and BCD output, ICL7135, is chosen to build the electromotive force measurement circuit. ICL7135 has input resistance as high as 1000MΩ, which affect little on the measured circuit [5]. As ICL7135 is a kind of AD converter using double integral circuit, we use the method of counting the clock of integral phase to get the voltage value. As Figure.4 shows, the ″busy″ output of ICL7135 remains high during the integral phase. The time of integration phase is constant, while the voltage is proportion to the time of anti-integration phase. So as long as the time of integration is acquired, the input voltage is obtained too.

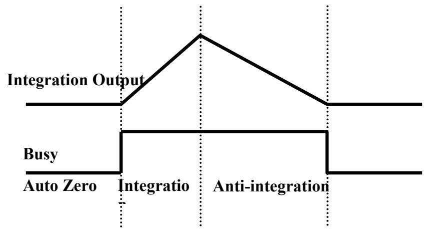

Figure 4. The ″busy″ pin output of ICL7135

Because of the impact of noise caused by wire and temperature shift, the value of electromotive force is not quite stable. So it is necessary to import a smooth filter here. We use a mixing filter method here to obtain relatively smooth results, as Figure.5 shows. The first-order filter with high coefficient will raise the sensitiveness and the mean filter will raise the smoothness as well.

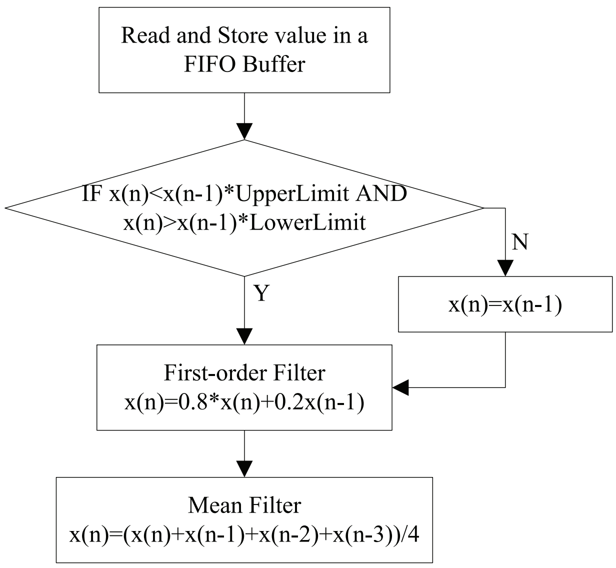

Figure 5. The flow chart of filter of electromotive force value

B. temperature measurement unit

The temperature measurement sensor used here is K-type thermocouple, so MAX6675 was chosen as the key component of the circuit. It performs cold-junction compensation and digitizes the signal from thermocouple.

The data is output in a SPI bus, which can be easily connected with MCU [6].

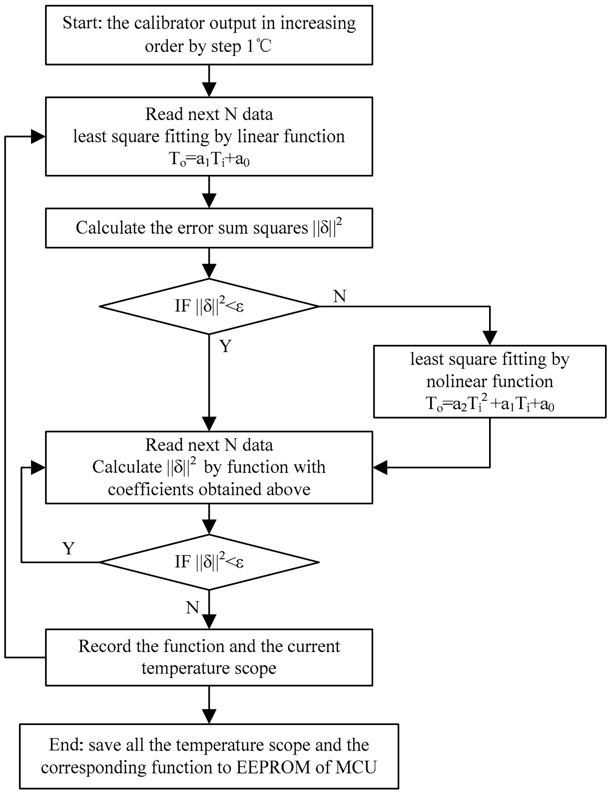

Figure 6. The flow chart of calibration algorithm

A lot of methods were adopted in circuit to achieve better measurement result, such as placing ceramic bypass capacitor to the supply, placing a large ground plane in the PCB. However, the accuracy of the MAX6675 is susceptible to noises and self-heating, a useful calibration scheme should be applied [7-8]. As shown in Figure.2, a PC and a Fluke5520A calibrator which can simulate the output signal of thermocouple are used for calibration. Firstly the computer sends the output command to the calibrator by small step continually. At the meantime the computer send the current value to MCU for further process. Secondly the output of calibrator connected with the input of MAX6675 will lead to a different value obtained in the MCU. Obviously the value of MAX6675 output should be calibrated to the standard value generated by the calibrator. Two different ways were considered to solve this problem. One is making a look-up table in the ROM of MCU after all the data was scanned, but it is not a good solution because the data amount is too large for storing and the MCU need to be programmed twice. The other is using curve-fitting technique by means of arithmetic operations that we decided to adopt.

Firstly all the possible temperature was scanned and then a data table of standard value and real value was made. It was found that the output of 6675 is not always linear in the whole temperature range, so it is not a good way to make a linear function approximation with all the data. To get a more precise curve-fitting, a piecewise function approximation algorithm using linear and nonlinear function was proposed. The algorithm flow is described as Figure.6 shows. The coefficients N and ε are very important to the algorithm, over-high N or ε may cause the curve-fitting be imprecise, while over-low N or ε may lead to too many function sections to handle. After repeated experiments, we choose $N = 10$ and $\varepsilon = 2.5$ for this algorithm. The calibration start from 0℃ to 1024℃, which is the measurement range of MAX6675. T_i is the output temperature of MAX6675 and T_o is the calibration temperature. Figure.7 and Table I shows the calibration results.

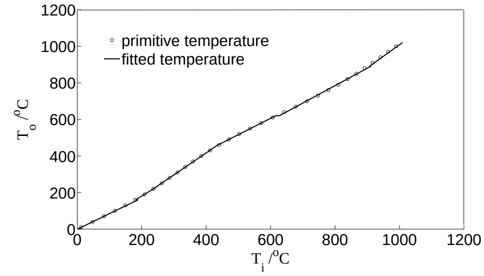

Figure7. The curve-fitting result of calibration

As Table I shows, the whole curve is divided into several parts by different linear or nonlinear function. Then all the important calibration information, including temperature range and function coefficients, will be saved in the EEPROM of MCU for future use in the measurement phase.

C. interactive units

To interact with the users, the matrix keyboard and LCD are used in the part of user interface which are easy to input and plain to show measurement results. Every $P_{H_2}^{II}$ value from the keyboard input can be saved in the SD card for the next measurement. All the measurement and calculation results will be saved in the card in plain file format. Results of every measurement occupy an independent file ordered by timestamp, which are very easy for further processing in the PC.

IV. RESULTS AND CONCLUSION

After precise calibration, this instrument was used in the hydrogen content measurement of a steel sample which is 16Mn rare-earth steel produced by the institute of metal research of chinese academy of sciences. After automatic and continuous measurement, the data saved in the SD card was taken out for analysis. Figure.8 shows the relation curve of temperature and $P_{H_2}^{II}$ value.

The experiments and tests show that the system has many good features. Its integrated circuit design makes it very portable to use without the assistance of any other equipment. The $P_{H_2}^{II}$ value can be input at all times when needed, which make it easy for changing the referring material for measurement. The system can show and save temperature, electromotive force and hydrogen content simultaneously. All the measurement data have been processed or calibrated before use, which make the measurement results more precise. All these features show that the system has a potential future in the hydrogen content measurement in metallurgical enterprises.

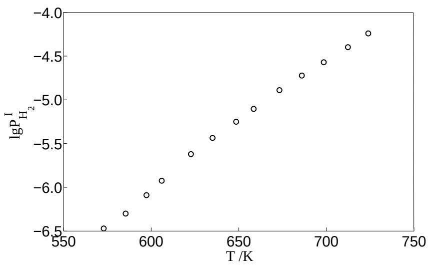

Figure8. The relation curve of temperature and $P^{II}_{H_2}$

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TABLE I. THE CURVE-FITTING FUNCTION RESULT OF CALIBRATION

Temperature Range (℃)	Fitted Function	Error Sum Square
0-160	T_o=1.17T_i+1.10	2.334
161-230	T_o=0.93T_i+32.64	2.017
231-460	T_o=0.83T_i+52.55	1.983
461-620	T_o=1.12T_i+78.51	1.322
621-890	T_o=1.05T_i-23.09	2.121
891-1024	T_o=0.0001T_i^2+0.59T_i+294.02	1.519