Research on Calibration Method of Temperature Field for Large Sealed High Temperature Equipment

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Abstract. Large sealed high temperature equipment usually refers to heat treatment equipment with a volume of more than 0.15m³, sealed structure and internal heating and constant temperature function. It is widely used in fields such as biochemistry, chemical pharmacy, medical and health, agricultural research, automobile and military industry. It’s a very important processing equipment. The accuracy of temperature field and vacuum sealing directly affect the quality of products. Taking the hot-pressing tank as an example, the furnace temperature tracker and wireless pressure module are used to calibrate it on the spot. According to the calibration results of temperature field distribution and temperature field performance, whether it meets the process requirements is analyzed.

1. Introduction
The large-scale sealed high-temperature equipment generally refers to thermal treatment equipment with volume over 0.15m³ [1], sealed structure, internal heating and constant temperature functions. Some devices are provided with vacuum pressure pump to create pressure or vacuum operating environment. It is an extremely important processing equipment which has been widely used in research fields such as biological chemistry, chemical and pharmaceutical engineering, health care, agricultural research, automobile and military industry. The product quality directly depends on the accuracy of thermal field and vacuum tightness. [2] However, nowadays, there is not a uniform measurement method for thermal field in China. Therefore, based on related standards [3], furnace temperature tracker and wireless pressure module are used to calibrate uniformity of thermal field, temperature deviation and fluctuation, vacuum and pressure tightness in working area, providing technical bases for industrial production and scientific research. [4]

2. Equipment under test
We take military autoclave for an example. Autoclave is a set of system equipment [5] that can realize timing sequence and real-time online control for process parameters such as temperature, pressure, vacuum and cooling, therefore, being an important processing equipment in many industries. See Figure 1 for its structure. It is mainly constituted by tank, sealing device, heating system, pressure system, automatic control system and safety mechanism. The tank is designed with length of 7m and height of 3.5m, of which internal working area is similar to shape of housing, being a 6m×Φ3m horizontal cylinder. Such working area is set with constant temperature range from ambient temperature to 400℃, general working pressure of 0.3MPa and top working pressure of 1.5MPa. Besides, the temperature uniformity is ±2℃ and deviation of control pressure does not exceed 10kPa.
3. Measurement devices

3.1. Furnace temperature tracker

The measurement device is constituted by furnace temperature tracker, insulated box, phase change module, sheathed thermocouple and wireless transmission module. See Table 1 for technical indexes.

Table 1. Technical indexes for components of temperature measurement devices [6]

| Name of components                  | Model       | Technical indexes                                                                 |
|-------------------------------------|-------------|----------------------------------------------------------------------------------|
| Furnace temperature tracker         | PTM1020     | Size: 200mm×98mm×20mm; 20 channels; measurement accuracy: ±0.3℃; sampling interval: Min. 0.5s |
| Thermal box                         | Phoenix TS02| Size: 550mm×400mm×300mm; Upper operating temperature: 1100℃                     |
| Phase change module                 | Phoenix PCA | Size: 280mm×145mm×75mm                                                            |
| Sheathed thermocouple               | K           | Length: 5m; outer diameter: 1mm; range of measurement: -40℃~1100℃; grade of accuracy: Grade 1 |
| Wireless transmission module        | TXR-1000    | Transmission frequency: 2.4Ghz; maximum transmission distance: 150m                |

The insulated box is designed with high-temperature resistant and stainless steel housing and filled with metallic oxide, fiber and high-temperature fabrics, porous and thermal insulation materials with thickness of 150mm. The internal groove is placed with metallic phase change module, while the box is fastened by two parts. When the buckle is tightened, sheathed thermocouple can extend out of the box from gaps on both sides. Due to heat absorption function, materials in phase change module will change from solid-state phase to liquid state and absorb thermal energy [7] under about 58℃. Besides, temperature will remain for a certain period to extend measurement time for furnace temperature tracker under high-temperature environment as much as possible. See Figure 2 for picture of thermal box and phase change module. The maximum operating temperature of monomer of furnace temperature tracker is 80℃ [8]. See Figure 3:
In addition to above hardware equipment, supporting testing software is also provided, in which corrected temperature values of calibrated sheathed thermocouple can be entered, and 20 channels can be calibrated one by one as per each temperature point.

3.2. Wireless pressure module
TMI wireless pressure module is used to measure pressure parameters. See Figure 4. For long-term operation, the module is measured with temperature range of (0–140)°C, measurement range of 3kPa–1.5Mpa, allowable error of ±1.2kPa, and over 40,000 stored data. [9] Besides, its tolerance does not exceed one fifth of the equipment under test and measurement accuracy satisfies with requirements. The data are recorded under sealed working environment and will be read via software system after test.
4. Methods of measurement

4.1. Measurement positions

Autoclave is provided with three internal separators on which uniform holes are designed. It is generally used for storing workpieces and can be used as temperature measurement rack when its temperature field is measured. Based on size and shape of internal working area in autoclave and temperature measurement position, in principle, temperature measurement positions shall be uniformly distributed in working area of autoclave, while measurement terminal of thermocouple does not contact with autoclave body or heating elements, including two important positions, 1. Geometric center of working area; 2. Measurement terminal of temperature control thermocouple in autoclave.

See Figure 5 for positions of insulated box and wireless pressure module. The wireless pressure module is positioned in the geometric center, while insulated box is positioned in the interlayer for distribution of sheathed thermocouple. Based on furnace mouth, measurement positions include “front”, “middle” and “rear” face and “front” and “rear” distributions are same. See Figure 6 for distribution position of each face and distance between two points. Position 7 refers to position of measurement terminal for temperature control thermocouple and Position 11 refers to geometric center of working area for furnace body. [10]

![Figure 5. Diagram of measurement positions](image1)

![Figure 6. Distribution diagram of measurement positions](image2)
4.2. Measurement procedure
The distribution of thermal field of autoclave is measured under unloaded condition. The measurement temperature is set to upper limit of 400℃. The upper limit of heat resistance for wireless pressure module is 140℃, which is far lower than measured temperature of thermal field. Therefore, automatic control system is closed and changed to manual mode. Besides, measurement for pressure indexes shall be executed firstly and then temperature shall be controlled independently to conduct measurement for thermal field.

According to operation of equipment, it will take 2h for autoclave to achieve thermal balance from ambient temperature to 400℃ and will take 3h from 400℃ to ambient temperature. Autoclave is provided with internal cross-ventilation equipment. The temperature measurement equipment shall be placed under high-temperature environment for measurement, so that measurement time shall be strictly controlled in order to prevent damage of furnace temperature tracker under high temperature. According to specification, for temperature measurement equipment constituted by various components, upper limit of temperature for normal operation is 1100℃ and duration does not exceed 30min. The high-temperature resistant time for measurement equipment is achieved through weighted calculation for five conditions as below: 1. Maximum temperature; 2. Time of duration; 3. Average temperature; 4. Working environment (e.g., carburizing atmosphere or static air); 5. Other factors such as handling of devices. When phase change module operates with low speed in convection air environment with maximum temperature of 400℃ and average temperature of about 240℃, normal operating time does not exceed 6h, so that temperature measurement time shall be strictly controlled within 1h.

We take wireless pressure module out after pressure measurement; put measurement devices in autoclave and complete wiring for sheathed thermocouple as required; start online and real-time data acquisition after heating up and maintaining constant temperature for 2h and set acquisition interval to 3min/times. After data acquisition, temperature is declined, and measurement equipment is taken out when requirements are complied with.

5. Measurement results
After excluding abnormal values [11], data collected for 1h are divided into 20 groups with 20 values in average for each. See Table 2 for distribution of thermal field of autoclave after calculation of average value.

| Setting temperature /℃ | Display temperature /℃ | Actual average value/℃ |
|------------------------|------------------------|------------------------|
| 400.0                  | 400.0                  |                        |
| Position 1             | Position 2             | Position 3             | Position 4 | Position 5 |
| 401.0                  | 400.9                  | 400.4                  | 400.7      | 400.7      |
| Position 6             | Position 7             | Position 8             | Position 9 | Position 10 |
| 400.7                  | 400.6                  | 400.8                  | 400.9      | 400.7      |
| Position 11            | Position 12            | Position 13            | Position 14 | Position 15 |
| 400.3                  | 400.5                  | 400.8                  | 400.8      | 400.7      |
| Position 16            | Position 17            | Position 18            | Position 19 | Position 20 |
| 400.8                  | 400.6                  | 400.4                  | 400.4      | 400.5      |

For above data, according to formula (1) & (2), uniformity of furnace temperature “$\Delta \theta_+ “$ & “$\Delta \theta_- “$ can be calculated; according to formula (3) & (4), stability of furnace temperature “$\delta_+ “$ & “$\delta_- “$ can be calculated; according to formula (5) & (6), stability of furnace temperature “$\Delta t_+ “$ & “$\Delta t_- “$ can be calculated as below:

$$\Delta \theta_+ = t_{pmax} - t_C$$

(1)
\[ \Delta \theta_+ = t_{p_{\text{max}}} - t_C \]  
\[ \Delta \theta_- = t_{p_{\text{min}}} - t_C \]  

Where,
- \( t_{p_{\text{max}}} \) refers to maximum temperature value for each measurement position (unit: °C);
- \( t_{p_{\text{min}}} \) refers to minimum temperature value for each measurement position (unit: °C);
- \( t_C \) refers to actual temperature for geometric center (unit: °C);

\[ \delta_+ = t_A - t_{c'} \]  
\[ \delta_- = t_i - t_{c'} \]  

Where,
- \( t_{c'} \) refers to average of indication values for geometric center (unit: °C);
- \( t_A \) refers to maximum value larger than \( t_{c'} \) measured at geometric center (unit: °C);
- \( t_i \) refers to minimum value lower than \( t_{c'} \) measured at geometric center (unit: °C);

\[ \Delta t_+ = t_{p_{\text{max}}} - t_b \]  
\[ \Delta t_- = t_{p_{\text{min}}} - t_b \]  

Where,
- \( t_b \) refers to nominal temperature (unit: °C);

The maximum temperature difference in furnace is calculated through subtracting minimum value by maximum value measured at each temperature measurement position during each measurement cycle. The measured pressure value is indicated with average value and tightness of autoclave is determined upon selected pressure fluctuation within 5min. [12] See Table 3 for results.

| Uniformity of furnace temperature /°C | Stability of furnace temperature /°C | Deviation of furnace temperature /°C | Maximum temperature difference in furnace /°C | Measured pressure value /kPa | Tightness of tank /kPa |
|--------------------------------------|--------------------------------------|--------------------------------------|-----------------------------------------------|-----------------------------|----------------------|
| \( \Delta \theta_+ \)                | \( \Delta \theta_- \)                | \( \delta_+ \)                       | \( \delta_- \)                               | \( \Delta t_+ \)             | \( \Delta t_- \)     |
| 0.6                                  | 0.0                                  | 0.1                                 | -0.1                                         | 1.0                         | 0.2                  |
|                                      |                                      | 0.8                                 | -0.9                                         | 0.8                         | 10.0                 |

6. Conclusions

According to above calibration results, for distribution of thermal field of such autoclave, temperatures of top layer and bottom layer are higher than middle layer and temperature of top layer is the highest. For front, middle and rear test faces, temperature if highest for front face near bottom of autoclave and lowest for face near the door. The uniformity and deviation of furnace temperature comply with requirements; stability of furnace temperature is good; tightness and pressure values also vary in reasonable range.

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