# The investigation of collagen fiber thermal deformation analyzer

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**Abstract**

The determination of 3 g constant tension and accuracy are the two main difficulties in detecting device of shrinkage temperature(Ts). The collagen fiber thermal deformation analyzer TMAF-YD4 has been developed to solve these problems based on the combination of non-contact displacement visual sensor and natural weighting method. This device could meet the requirements of QB/T2713—2005 and significantly improve the accuracy of displacement detection (0.01 mm). Furthermore, the uniformity of the heating temperature of medium was increasing by the method of composite control and stirring. Effectively The accuracy and credibility of detection data was effectively enhanced by the improvement of sample hanging and standard sampler. Modern processing methods such as real-time display and output of a large amount of data on the host interface may provide strong technical supports for further professional research and analysis to meet different kinds of users. The TMAF-YD4 has been utilized in the market for one year with good feedbacks, and its application has been extended to biomedical engineering fields such as heart valves.

Keywords: Shrinkage temperature, 3-gram constant tension, temperature uniformity, sample fixation, data processing

1. **Introduction**

Leather with special structural based on collagen fibers has played an important role in people's daily life. The shrinkage temperature is one the main factors to determine the moisture and heat resistance stability, which has significant guidance on the construction of tanning processes. Thus, the development of standard detection instruments for Ts with high speed and accuracy is important for the development of sustainable leather industry. According to the national standard of GBT4689.8-1984 [1] and international standard of ISO3380-2002, the Ts value of leather samples was defined as the temperature of heating liquid medium once the sample begins to shrink and deform to a degree of 0.3% with a heating increase of 2 °C/min at the state of 3 g constant tension. The schematic diagram of the working principle and the original device were shown in Fig. 1a-b. However, the Ts parameter detection device with appropriate cost and convenience has not been served in the market.

Many researchers have explored detection device for users, such as PS-83(Fig. 1c) and STD114(Fig. 1d) detection device from shandong textile research institute and SATRA in the UK, respectively. There are certain difficulties in the promotion and application of these devices due to insufficient detection conditions,

low accuracy and complex operation during the detection process. Interestingly, the MSW-YD4 leather shrinkage temperature tester (Fig. 2) was developed 10 years ago for solving these problems [2-4], which is invented by sunshine electronics research institute of shaanxi university of science and technology and institute of creative materials. This device was popular among schools and industries for high cost-effectiveness and easy operation and granted for shaanxi province jey new product title and third prize for science and technology progress. The whole device was consisted of three parts: host, upper computer, leather sampler.

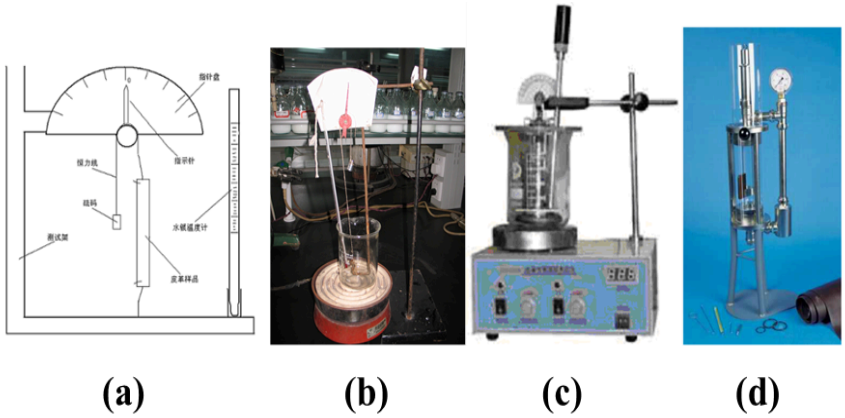



Fig. 1 The principle of Ts parameter detection and related detection devices




Fig. 2 MSW-YD4 Leather shrinkage temperature tester

A microcontroller is used as the control core for the MSW-YD4 shrinkage temperature measuring instrument. The constant temperature rise of the liquid heating medium at 2 °C/min is effectively controlled through a closed-loop switch, and the detection conditions are implemented by providing the sample in a constant tension state of 3g through spring compression in the sensor. The data signals of the shrinkage deformation of the sample after heating and the temperature of the heating medium were collected using specially developed Hall displacement sensors and AD590 temperature sensors, respectively. Therefore, when compared to traditional similar detection instruments, it has the following technical advantages:1. high precision measurement (displacement accuracy≤0.2 mm, temperature accuracy≤0.1 °C, and the heating rate is within 2±0.2 °C /min); 2. efficient detection (four samples can be tested simultaneously at once); 3. multifunctional output (display the change curves of length shrinkage deformation and temperature rise of the four samples throughout the entire testing process); 4. simple and safe operation (upper limit of heating medium temperature and automatic locking). Based on these advantages above, the

MSW-YD4 systolic temperature measuring instrument has received unanimous recognition from different users. Its current application has expanded to biomedical engineering fields such as blood vessels and heart valves.

2. **Main technical issues of MSW-YD4**

### 2.1 Key technology

After careful analysis of the relevant content of the detection standards, the key technology can actually be summarized as the implementation of two detection conditions (1. a 3 g constant tension state of the sample; 2. 2 °C/min constant heating speed) and the real-time collection and processing of two data (1. Accuracy data timely collected of shrinkage deformation of samples; 2. uniformity of temperature field in heating medium and typicality of sampling points).

### 2.2 Main problems

With the development of social demand and the progress of science and technology, the technical defects of the MSW-YD4 shrinkage temperature measuring instrument have become increasingly prominent. Compared with the above analysis, the main manifestations are incomplete detection conditions, low measurement accuracy, and single data processing.

#### 2.2.1 Detection conditions

The Hall displacement sensor (Fig. 3a) can theoretically achieve a constant tension detection condition of 3g for the sample by a pressure spring. However, the length of the samples among the testing process is greater or less than the standard size of 50 mm because of the plasticity of leather samples. Thus, the tension of the sample cannot maintain the detection conditions required by the standard for 3g constant tension according to Hooke's law (Fig. 3b). The stability of the system's operation is improved by The closed-loop control switch type electric heating method. Fluctuations in the temperature of the heating medium in the time domain during the heating process are also brought. Heating media with high viscosity coefficients such as glycerol without stirring can lead to the generation of temperature gradients in the three-dimensional space region. As a result, there are doubts on the non-uniformity of temperature distribution in the airspace and the representativeness of temperature collection points.

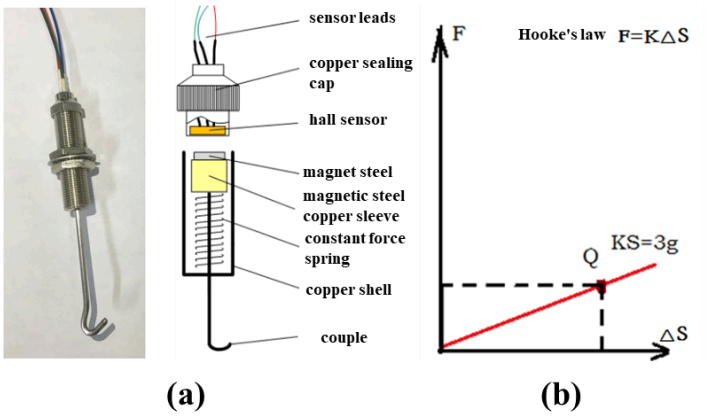



Fig. 3 (a)Structure of Hall displacement sensor, (b) Hooke’s law

#### 2.2.2 The accuracy of measurement

The Hall type displacement sensor used for shrinkage deformation measurement has problems such as low accuracy, analog signal output, nonlinearity, and "magnetic saturation", which inevitably leads to significant displacement measurement errors. The AD590 temperature sensor has problems with small

temperature measurement range and nonlinearity, making it difficult to achieve high-precision temperature measurement.

### 2.2.3 Data processing

The Ts value is displayed in the host, and 485 wired communication mode and dedicated software interface are used to indicate five curves of the shrinkage deformation curve and temperature rise curve of the four samples during the entire test process, which are seemed to be simple. Conventional contact displacement sensors can achieve the accuracy of 0.01 mm, however it cannot maintain the 3 g constant tension with the sample during the testing process because of touching with the fixed end. The high-precision laser displacement sensor LTP080, which currently meets the usage requirements (detection range: ±15mm, accuracy: 0.012mm) cannot be applied in the market for high price (15000 RMB).

## 3. **Key technologies of TMAF-YD4**

On the basis of the MSW-YD4 leather shrinkage temperature measuring instrument, the TMAF-YD4 collagen fiber thermal deformation analyzer has been successfully developed to meet the detection conditions of the sample under 3g constant tension [7,8]. The temperature field uniformity in the spatiotemporal domain has been greatly improved (the temperature difference between the upper and lower ends of the sample is ≤ 0.4 °C), and the displacement detection accuracy is ≤ 0.01mm [5,6]. The data can be displayed in the screen with three modes: curves, key node data, and graphics. It has received positive reviews and recognition from users.

### 3.1 Achievements of detection condition of 3 grams of constant tension and 0.01mm displacement accuracy

As shown in the left figure in Figure 4a, the sample is vertically fixed between the upper and lower hooks in a hanging hole manner. The upper hook set on the detection board is a static hook, the lower hook is a dynamic hook, and it is integrated with the exported slide bar. The top of the slide bar is marked and always in the camera's view. Once in the detection state, the sample will automatically dive into the heating medium after the heating container rises. For samples with length of 50 mm, the shrinkage deformation range is usually within 25 mm. An 8-million-pixel camera is used to capture targets within a 25*25mm field of view that each pixel represents a linear displacement size of<0.01mm. The actual displacement accuracy is ensured to be greater than 0.01mm due to the reliable and stable transmission of digital signals without interference and conversion. During decades of experiments, it has been found that signal-to-noise ratio would decrease due to wastes in the white light source board during long-term use, which interfere the accuracy of sample shrinkage deformation data collected by the camera. Thus, the white light source board was replaced with the black light source board. Furthermore, an ultraviolet excitation luminescence and phosphor were added in this system to avoid the effect of wastes (Fig. 4b).

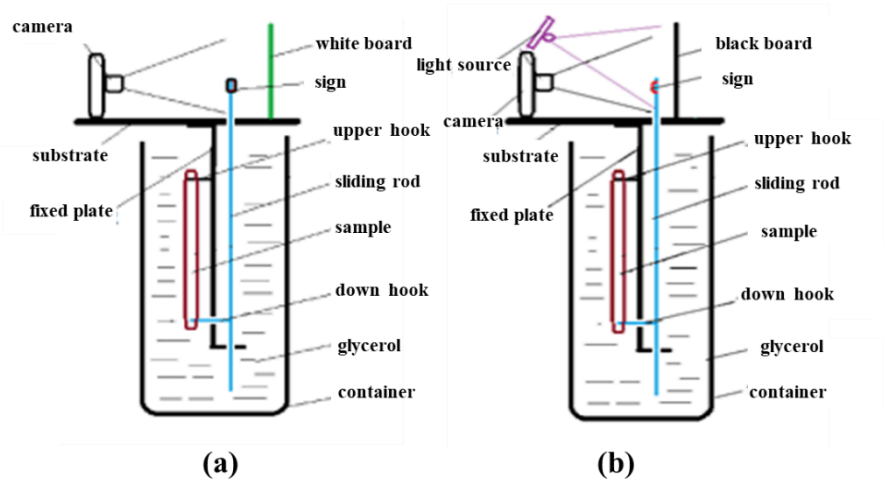



Fig. 4 The structure of TMAF-YD4

### 3.2 Improved temperature uniformity and data accuracy of spatiotemporal heating media

The combination of a dual load and a loop composite switch control method is used to reduce the temperature fluctuation at 1/4 of the original, improving the temperature uniformity in the time domain, as shown in Figure 5. The heating method of the liquor was transformed from line heating to surface heating. Uniformity of liquid space temperature is guaranteed by natural circulation and stirring. The two heating loads are set in contact with the bottom of the cup, ensuring better heat transfer and uniformity of the heated liquid in three-dimensional space. Temperature field uniformity of heating media such as glycerol with high viscosity coefficient is ensured by increasing stirring. The temperature sensor is set in the middle of the sample length to concern the height direction of the vertically specimen. The optimal location within the horizontal plane of the heating medium space is not considered, causing doubts about the typical of temperature data. Therefore, the position of samples is changed and length of couple is increased to decrease doubts as shown in Fig.6.

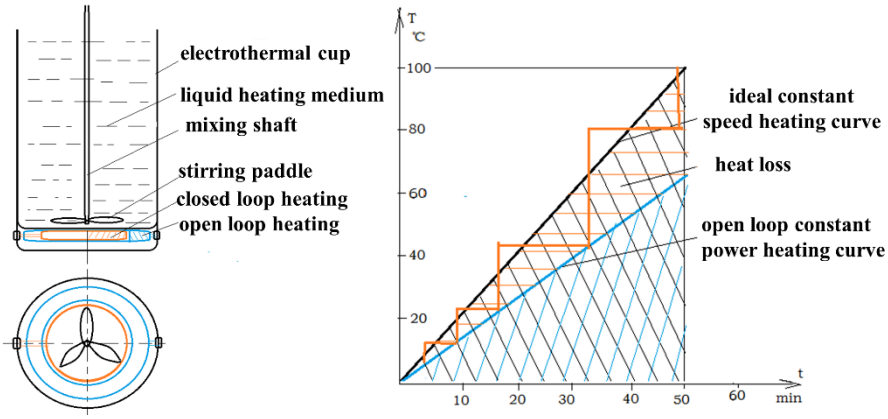



Fig. 5 The principle of improving the uniformity of liquid heating in the airspace

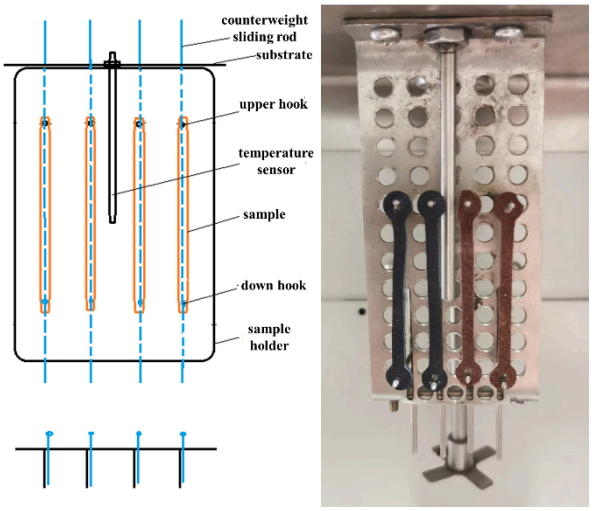



Fig.6 Schematic diagram for selecting temperature collection points

4. **System composition and performance characteristics**

The TMAF-YD4 collagen fiber thermal deformation analyzer is consisted of four parts: the host, sampler, vacuum infiltration device, and upper computer software, as shown in Figure. 7. The sampler is used to make test samples with the standard knife mold. A double hole sample is made from the tested leather at once according to the requirements for further testing. Manual samplers are suitable for ordinary laboratories, while electric ones are more suitable for the production of large batches of samples (such as student experiments or other situations). The vacuum infiltration device is prepared for adjusting the condition of solid sample according to the technical requirements of national standards. The host is mainly composed of a data acquisition unit and a control display unit, completing basic functions such as data acquisition and processing, as well as display and output. It is the main equipment in the system, as shown in Figure 8. The data collection department is composed of a data collection box, sample holder, non-contact displacement sensor, temperature control system, automatic lifting platform, and stainless steel heating container. Its function is to ensure that the length of the sample and the temperature of the surrounding medium are accurately collected and provide to the data processor in real-time. In order to obtain the changes (host interface curves, key nodes, and graphics) in length and temperature of all specimens, a rich and user-friendly host touch screen display interface is designed. The host is connected with a computer to get the real-time data and these data can be displayed by curves, data, and graphs (Fig. 9).

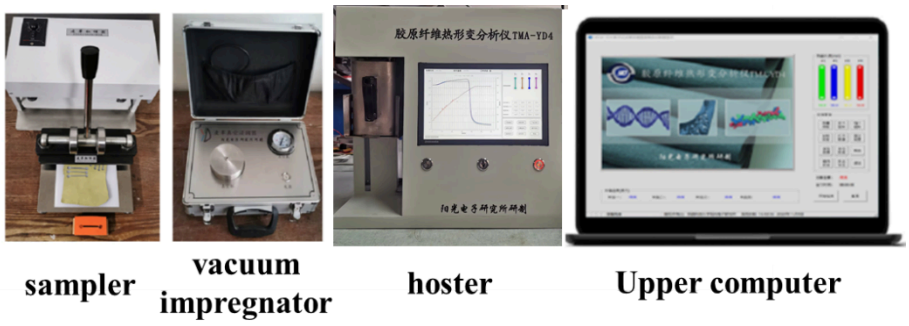



Fig.7 Four components of the TMAF-YD4 collagen fiber thermal deformation analyzer system

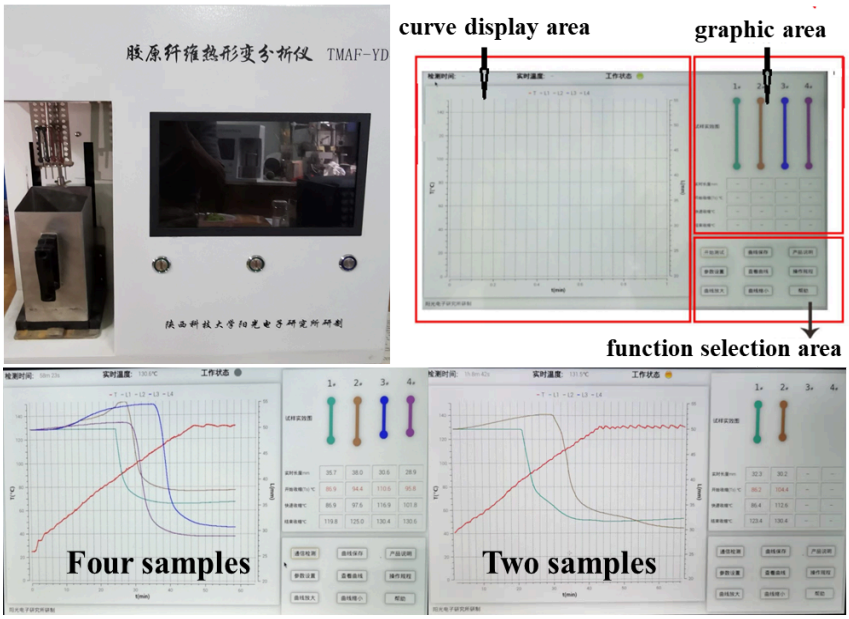
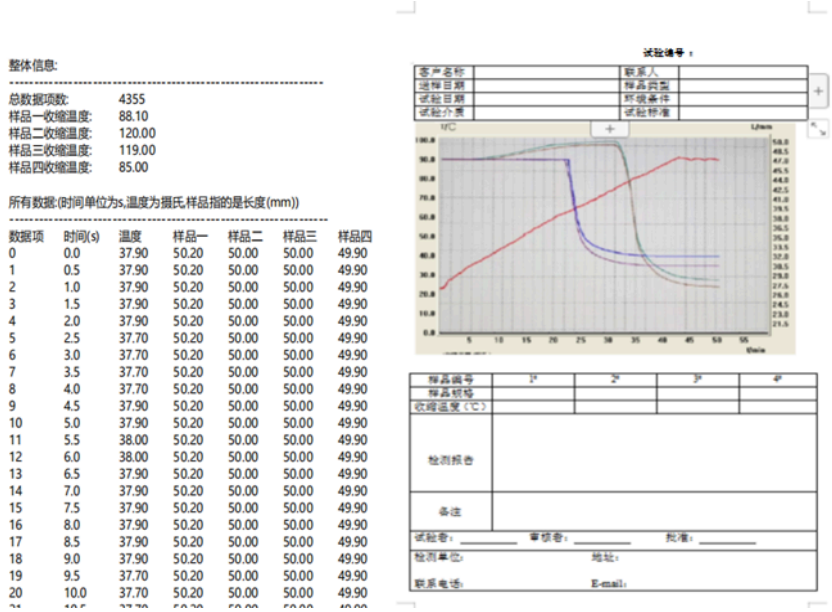



Fig.8 Data display



整体信息:

总数据项数: 4355
样品一收缩温度: 88.10
样品二收缩温度: 120.00
样品三收缩温度: 119.00
样品四收缩温度: 85.00

所有数据:(时间单位为s,温度为摄氏,样品指的是长度(mm))

| 数据项 | 时间(s) | 温度 | 样品一 | 样品二 | 样品三 | 样品四 |
|---|---|---|---|---|---|---|
| 0 | 0.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 1 | 0.5 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 2 | 1.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 3 | 1.5 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 4 | 2.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 5 | 2.5 | 37.70 | 50.20 | 50.00 | 50.00 | 49.90 |
| 6 | 3.0 | 37.70 | 50.20 | 50.00 | 50.00 | 49.90 |
| 7 | 3.5 | 37.70 | 50.20 | 50.00 | 50.00 | 49.90 |
| 8 | 4.0 | 37.70 | 50.20 | 50.00 | 50.00 | 49.90 |
| 9 | 4.5 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 10 | 5.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 11 | 5.5 | 38.00 | 50.20 | 50.00 | 50.00 | 49.90 |
| 12 | 6.0 | 38.00 | 50.20 | 50.00 | 50.00 | 49.90 |
| 13 | 6.5 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 14 | 7.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 15 | 7.5 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 16 | 8.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 17 | 8.5 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 18 | 9.0 | 37.90 | 50.20 | 50.00 | 50.00 | 49.90 |
| 19 | 9.5 | 37.70 | 50.20 | 50.00 | 50.00 | 49.90 |
| 20 | 10.0 | 37.70 | 50.20 | 50.00 | 50.00 | 49.90 |

Fig.9 Examples of data report

There some advantages in the TMAF-YD4 collagen fiber thermal deformation analyzer. During the testing process, the sample is always in an ideal state of constant tension of 3 grams (non-contact visual sensor combined with natural weight method), and the temperature difference between the upper and lower ends of the sample is ≤ 0.3 °C. The data error of displacement accuracy is less than 0.01 mm and temperature accuracy is no more than 0.5 °C. The environmental temperature, sample length, and other parameters of the entire thermal deformation process of the four samples are displayed in real-time on the host interface through curves, data, and images. Each group of six data is uploaded to the upper computer at a frequency of 2 sets/second. Four samples can be tested at the same time and standard testing report is provided. The heating rate can be adjusted such as 0.5, 1, 2, 3 °C/min. During the detection process, t, T, L1, L2, L3, and L4 data are displayed in real-time on the host interface in three ways: curves, numbers, and graphics. Comparison of thermal deformation curves of four samples in entire process is displayed.

5. **Conclusion**

The goal of 3 g tension in sample and improvement of displacement detection accuracy are achieved by a non-contact displacement sensor combined with natural configuration method for visual technology during testing process. The signal-to-noise ratio and reliability are increased by emitting monochromatic light. A dual load structure and composite control method is used to control the heating rate at 2 °C/min. While the uniformity of heating medium is maintained by stirring and optimization of collections. The TMAF-YD4 collagen fiber thermal deformation analyzer can meet the requirements for Ts detection with high credibility and accuracy. The host is connected with the terminal computer so the exhibition of data is diversification. The TMAF-YD4 collagen fiber thermal deformation analyzer has been used in the laboratory of Sichuan university and Shaanxi university of science and technology for more than one year. Moreover, it has been expanded the field of biomedical tissue engineering materials - the detection of wet heat contraction temperature Ts of heart valves such as Shanghai Newmai Medical and Chengdu Newmai Biological. It can combine with MSW-YD4 leather shrinkage temperature tester to form a series of mid to high-end Ts detection and analysis instruments, assisting the development of the leather and biomedical engineering industries.

## 6. **Acknowledgements**

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