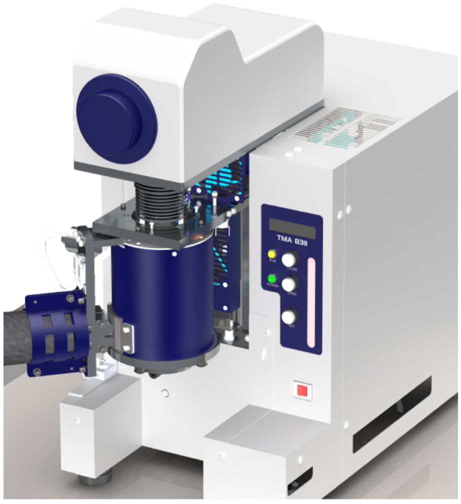
Thermomechanical analyzer with refrigerated cooling unit

Thermo plus EVO2 TMA8311/LR

A minimum temperature of −70°C is achieved using the refrigerated cooling unit equipped TMA



1. Introduction

In recent years, there has been an increasing demand for non-liquid nitrogen based cooling systems in thermal analysis because of environmental considerations and usability. Especially in differential scanning calorimetry (DSC), refrigerated cooling unit is often used. Using the Rigaku DSC, low temperature measurements from −90°C can be carried out. This indicates that refrigerated cooling can cover most DSC applications, and thus LN_2 cooling is only used for measuring a few limited materials.

Recently, Rigaku has developed the TMA8311/LR thermomechanical analyzer (TMA) with a refrigerated cooling unit. Previously, sub-ambient TMA measurement required LN_2 cooling, but the TMA8311/LR enables LN_2-free measurements thanks to the cooling system that achieves the lowest cooling temperature of −70°C, similar to the refrigerated cooling unit equipped DSC.

2. Product Features

2.1. −70°C to 600°C measurement temperature range

The cooling technique used for the Rigaku DSC systems has been adopted in the TMA8311/LR, which enables TMA measurement with the minimum temperature of −70°C. In addition, the highest temperature specification is 600°C. This wide temperature range expands applications, especially in the polymer field.

2.2. Effectiveness of cycle measurement

For measurements where the 2nd run result is important, using the TMA with refrigerated cooling unit allows cycle measurement with controlled cooling. This method eliminates the instability caused by thermal hysteresis during cooling and has dramatically improved its complex operation. Figure 1 shows the result of a cycle measurement of epoxy resin. In the initial heating process, a shrinkage occurred at 40°C due to the effect of thermal hysteresis of the sample. Then, in the cooling process and the second heating process, a change in expansion rate at 70°C due to glass transition can be confirmed. As the result, $\alpha_{1(30\text{–}50°C)}: 10.6\times10^{-5}$ (1/K) and $\alpha_{2(120\text{–}140°C)}: 18.8\times10^{-5}$ (1/K) can be calculated from the second heating process of a single measurement.

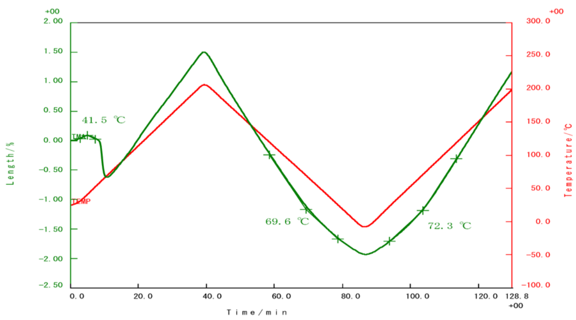


Fig. 1. Result of a cycle measurement of epoxy resin.

2.3. Improved rate stability with the new temperature control method

In TMA with refrigerated cooling unit, the existing control algorithm of heating/cooling rate has been reviewed and replaced with a new control algorithm, which minimizes the control delay that appeared at the start of a heating process or after a change in heating rate. The new control algorithm allows the temperature to promptly reach the specified heating/cooling rates.

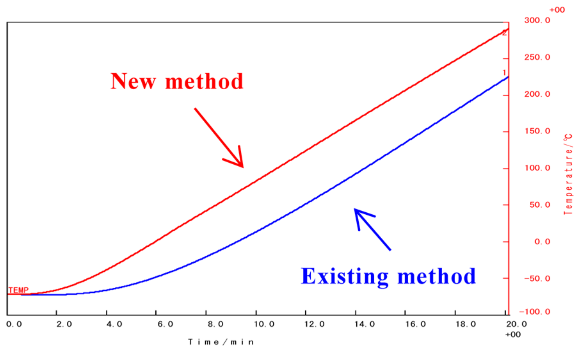


Fig. 2. Comparison of temperature change between existing and new controls (20°C/min.).

2.4. Versatility

The refrigerated cooling furnace of TMA8311/LR can be replaced with the standard electric furnace or LN_2 cooling furnace even by users. This refrigerated cooling unit is compatible with the existing Thermo plus EVO2 TMA and can be installed as an after purchase accessory.

2.5. Usability

Optionally, a synchronized power ON/OFF link unit is available. This unit enables to automatically shut down the TMA system, including the refrigerated cooling unit and the gas flow unit after the measurement ends. In addition, an auto-startup setting that will initiate the TMA at a preset time is also available, which helps save cooling time (1 hour) after the TMA turns on. There is no need to wait until the measurement is ready.

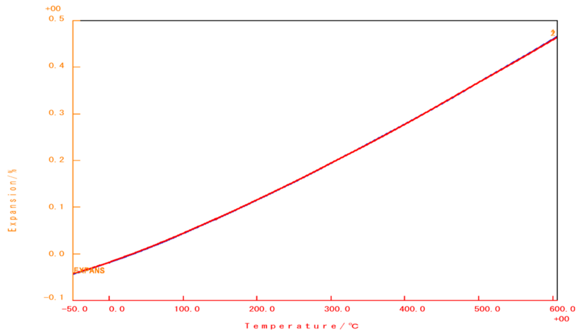
3. Applications

In the TMA with refrigerated cooling unit, the temperature uniformity within the furnace is improved through the isothermalizing tube and constant thermalization.

These improvements also increased the stability in thermal expansion measurements, compared to when using a standard electric furnace or LN_2 cooling system.

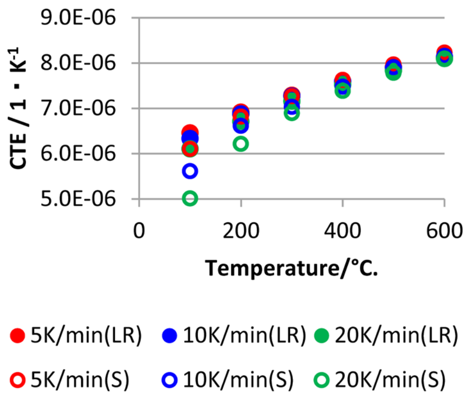
Figure 3 shows multi-plot of c-axis sapphire thermal expansion during heating and cooling processes at 5°C/min. The graph shows that nearly all the same plots are observed in both the heating and cooling processes. The average expansion coefficient from 30 to 600°C are 8.12×10^{-6}(1/K) and 8.17×10^{-6}(1/K) in the heating process and the cooling process, respectively; and their difference is 0.6%.

Figure 4 shows the comparison of the average expansion coefficient (base temperature: 30°C) of the c-axis sapphire measurement using the refrigerated cooling furnace (LR) and the standard electric furnace (S). With the refrigerated cooling furnace, the difference



Heating/cooling rate	Heating process	Cooling process	
5°C/min	8.12×10^{-6}	8.17×10^{-6}	(1/K)

Fig. 3. Multi-plot of c-axis sapphire's thermal expansion (base temp.: 30°C) in the heating and cooling processes with a rate of 5°C/min and the average coefficient of linear expansion between 30°C and 600°C.



Temp. (°C)	Refrigerated cooling furnace				Standard electric furnace			
	5 K/min	10 K/min	20 K/min	ΔCTE	5 K/min	10 K/min	20 K/min	ΔCTE
200	6.91	6.87	6.71	0.20	6.81	6.61	6.21	0.60
400	7.58	7.59	7.52	0.06	7.62	7.47	7.38	0.24
600	8.12	8.12	8.09	0.03	8.22	8.15	8.09	0.13

($\times10^{-6}$ 1/K)

Fig. 4. Average expansion coefficient of c-axis sapphire measurement with the refrigerated cooling furnace (LR) and standard electric furnace (S) with different rates of 5°C/min, 10°C/min, and 20°C/min.

in the average expansion coefficient of each heating rate is smaller compared to the standard electric furnace. This indicates that even with high heating rates, the isothermalization within the furnace of the TMA with refrigerated cooling unit has improved.

4. Conclusion

In the field of polymer, cycle measurement is also widely performed in TMA. However, the ease of use of TMA with refrigerated cooling unit has improved greatly compared to the liquid nitrogen-based cooling system.

Cooling measurements can be easily performed through the TMA with refrigerated cooling unit and it can be used in a wide range of applications in the future.