APPLICATION HANDBOOK THERMAL ANALYSIS

热分析应用手册

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药物和食品

PHARMACEUTICALS & FOOD

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Application Handbook
Thermal Analysis

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本应用手册提供精选的应用实例，由瑞士梅特勒－托利多热分析实验室采用相关的仪器进行实验，作者根据最新知识进行数据处理。

但这并不意味着读者无需用适合试样的方法、仪器和用途进行亲自测试。由于对实例的效仿和应用是无法控制的，所以我们无法承担任何责任。

使用化学品、溶剂和气体时，必须遵循常规安全规范和制造商或供应商提供的使用指南。

This application handbook presents selected application examples. The experiments were conducted with the utmost care using the instruments specified in the description of each application at METTLER TOLEDO Themal Analysis Lab in Switzerland. The results have been evaluated according to the current state of our knowledge.

This does not however absolve you from personally testing the suitability of the examples for your own methods, instruments and purposes. Since the transfer and use of an application is beyond our control, we cannot of course accept any responsibility.

When chemicals, solvents and gases are used, general safety rules and the instructions given by the manufacturer or supplier must be observed.

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序

热分析是仪器分析的一个重要分支，它对物质的表征发挥着不可替代的作用。热分析历经百年的悠悠岁月，从矿物、金属的热分析兴起，近几十年在高分子科学和药物分析等方面唤起了勃勃生机。

我国在20世纪50～60年代，科研单位、高校和产业部门为满足科研、教学和生产的需要，经历了从原理出发自行设计研制热分析仪器的艰苦创业阶段；30～40年前，先进的热分析仪器还只是在少数科研单位的测试中心才拥有，而随着我国综合国力的增强和对科研支持力度的加大，现已逐渐成为许多实验室的通用仪器，在科研和生产中起着愈来愈重要的作用。广大相关专业的科技人员为了更好地利用这些设备，迫切需要深入掌握热分析仪器及相关的基础和应用方面的知识。这套热分析应用系列丛书就是在这样的形势下应蕴而生的。

本书的基础数据主要是由瑞士的梅特勒－托利多(Mettler－Toledo)公司提供的，该公司是全球著名的精密仪器制造和经销商。早在1945年，就曾以首台单秤盘替代法天平而闻名于世。随后，又将其与加热炉结合，在1964年推出了世界上第一台商品化TGA/DTA热分析仪器。1968年又有TGA/MS联用仪和差示扫描量热仪(DSC)相继问世。40余年来，梅特勒－托利多一直是全球热分析仪器的主要供应商之一。现今具有包括DSC、TGA/DSC、TMA、DMA等完备的现代热分析仪器。近年取得的新进展有如：多星型热电堆DSC 2006年荣获美国R&D100奖，该奖项是每年颁发给当年在全球技术领域具有代表性新产品的开发者；2005年开发的随机多频温度调制DSC技术TOPEM™，能在一次实验中测定准稳态比热容，由热流与升温速率的相关性分析分别得到可逆、不可逆热流量和总热流量，以及反应(转变)过程与频率的关系。该公司最新推出的闪速DSC(Flash DSC)更是热分析仪器的一项创举，为人们观测物质在快速升降温时的变化提供了新视解。

《热分析应用手册》系统介绍了热分析在诸多领域的应用。这套丛书是汇集了梅特勒－托利多公司瑞士总部和梅特勒－托利多(中国)公司科技人员的智慧而潜心编著的。《药物与食品》是其中的一个分册，热分析在药物熔点、纯度测定和多晶型观测等方面有极其广泛的应用。食品安全备受人们关注，按药食同源说，许多食品具有药物作用，含有某些生物活性成分，热分析可测定食品的组成、推测食品的新鲜程度和保持期，热分析是十分有效的快速检测食品的手段。在上述方面本书均给出了典型的应用示例。

该分册的主要作者Jürgen de Buhr和Georg Widmann长期在梅特勒－托利多瑞士热分析实验室工作，是热分析技术应用方面的资深专家。

译者陆立明先生1985年在华东理工大学获得聚合物材料工学硕士，后在上海市合成树脂研究所从事聚合物研究开发工作12年(其中3年在德国柏林技术大学进修高分子物理)。加入梅特勒－托利多(中国)公司以来一直从事热分析的技术应用和管理工作。

本书的酝酿和出版正值我国改革开放30年，我国在世界的影响全面提升。据报道这一时期我国科技人员在国际期刊发表的论文数量迅速提高。曾有报道，2004年，中国科学院发表SCI论文较1998年增长115%，总量已约为德国马普学会的2倍；在国际各领域居前20位学术刊物上发表的高质量论文数量，已占全国同期总量的一半以上(见：科学时报，2009年7月3日第2版)。

本书的一个明显特点是以中英文对照的形式出版，这就为需要熟悉英语论文写作方法的读者提供了一种借鉴。

相信这套丛书的出版，将会对我国热分析技术的普及与提高起到重要的推动作用。

刘振海

2011年7月于中国科学院长春应用化学研究所

出版前言

《热分析应用手册系列丛书》是由梅特勒－托利多瑞士热分析实验室专家撰写的系列手册，包括《热分析应用基础》、《热塑性聚合物》、《热固性树脂》、《弹性体》、《食品和药物》、《逸出气体分析》和《认证》等分册。

这套书既注重实用性，又注重学术性。可以将它们作为应用手册查询，也可以作为实验指南，如帮助选择合适的热分析测试技术和方法、制备和处理样品，设定实验参数等。手册中的所有应用实例都经过认真挑选，实验方法经过精心设计，测试曲线重复可靠，数据处理严格谨慎，结果解释和结论推导科学合理。

这套手册面向所有用到热分析和对热分析感兴趣的教授、科学家、工程师和学生（特别是研究生），适合所有热分析仪器的直接使用者。

本书是《热分析应用手册系列丛书》之《食品和药物》分册。英文原稿为《药物》和《食品》两个分册，我们将它们合并为一册出版。

热分析技术在药物以及食品行业具有广泛的应用，例如药物活性成分和非活性成分的分析表征、食物蛋白质变性的分析表征、安全性分析、稳定性研究以及质量控制中的日常分析。

本书简要介绍了 DSC（差示扫描量热法）、TGA（热重分析法）、TMA（热机械分析）和 DMA（动态热机械分析）等热分析的主要技术；通过许多实例，多方面深入介绍和讨论了热分析在药物和食品方面的应用。

与其他大多数分册一样，本书以中英文对照方式出版。本书无论对热分析工作者，还是对热分析学习者，应该都有帮助。

这里要特别感谢刘振海教授为本书作序。他仔细审阅了本书全部书稿，并逐字逐句进行修改，使本书的质量得到了很大提高。

同时感谢东华大学出版社编辑为出版本套丛书作出的辛勤努力。

译文甚至原著中，有错误之处，恳望读者指正，以便能在再版时改正，不胜感谢。

陆立明

2011 年 7 月，上海

目　录

1 热分析概论 Introduction to Thermal Analysis

热分析是测试材料的物理和化学性能与温度关系的一类技术的总称。在所有这些方法中，样品受加热、冷却或等温温度程序控制。

测试可在不同气氛中进行，通常使用惰性气氛(氮气、氩气、氦气)或氧化气氛(空气、氧气)。有时在测试期间从一种气氛切换到另一种气氛。另一个可选择的参数是气体压力。

DSC 还可与能同步观察试样的仪器联用(DSC/显微镜法)，或用不同波长的光照射(光量热法)。

Thermal analysis is the name given to a group of techniques used to measure the physical and chemical properties of materials as a function of temperature. In all these methods, the sample is subjected to a heating, cooling or isothermal temperature program.

The measurements can be performed in different atmospheres. Usually either an inert atmosphere (nitrogen, argon, helium) or an oxidative atmosphere (air, oxygen) is used. In some cases, the gases are switched from one atmosphere to another during the measurement. Another parameter sometimes selectively varied is the gas pressure.

DSC can also be used in combination with instruments that allow the sample to be simultaneously observed (DSC microscopy) or exposed to light of different wavelengths (photocalorimetry).

1.1 差示扫描量热法(DSC) Differential Scanning Calorimetry

DSC 测量流入和流出试样的热流量。DSC 可用于研究物理转变(玻璃化转变、结晶、熔融和挥发化合物的蒸发)和化学反应的热效应，所获得的信息可表征样品的热性能和组成。此外，还能测定诸如热容、玻璃化转变温度、熔融温度、反应热和反应程度等的性质。

In DSC, the heat flow to and from the sample is measured. DSC can be used to investigate thermal events such as physical transitions (the glass transition, crystallization, melting, and the vaporization of volatile compounds) and chemical reactions. The information obtained characterizes the sample with regard to its thermal behavior and composition. In addition, properties such as the heat capacity, glass transition temperature, melting temperature, heat and extent of reaction can also be determined.

1.1.1 常规 DSC Conventional DSC

常规 DSC 采用线性温度程序，试样和参比物(通常只是空坩埚)以线性速率升(降)温，或经常将几个局部程序即所谓的程序段连接在一起，形成一个完整的温度程序。典型的 DSC 曲线如图 1.1 所示。测试开始时曲线的变化是由于初始的“启动偏移”(曲线 1)。在该瞬变区域，状态突然从等温模式变为线性升温模式。启动偏移的大小取决于试样热容和升温速率。试样中如果存在挥发性物质如溶剂，会观察到由于蒸发产生的吸热峰(曲线 2)，试样

Conventional DSC employs a linear temperature program. The sample and reference material (or just an empty crucible) are heated or cooled at a linear rate, or in some cases, held at a constant temperature (i. e. isothermally). Often several partial programs or so-called segments are joined together to form a complete temperature program. A typical DSC curve is shown schematically in Figure 1. 1. The change in the curve at the beginning of the measurement is due to the initial “startup deflection” (1). In this transient region, the conditions suddenly change from an isothermal mode to a linear heating mode. The magnitude of the startup deflection depends on the heat capacity of the sample and the heating rate. If volatile substances such as solvents are present in the sample, an endothermic peak (2) is

失重。可通过称量测试前后的试样质量和使用不同种类的坩埚得到关于这种峰的更多信息。与开口坩埚不同，完全密封的坩埚可防止试样的蒸发。无热效应的 DSC 曲线部分(曲线 3)，由于试样的热容通常线性增大，因而观察到称为“基线”的直线。熔融产生吸热峰(曲线 4)。最后，在较高的温度，开始分解(曲线 5)。实验所用吹扫气体的种类经常会影响发生的反应，尤其在高温下更是如此。

observed due to the vaporization; the sample loses mass. Further information on such peaks can be obtained by weighing the sample before and after the measurement and by using different types of crucibles. In contrast to open crucibles, hermetically sealed crucibles prevent vaporization of the sample. At the part of DSC curve with no thermal effects (3), the heat capacity of the sample increases normally linearly and therefore a straight line called “baseline” is observed. The melting produces endothermic peak (4). Finally, at higher temperatures, decomposition begins (5). The type of purge gas used in the experiment often has an influence on the reactions that occur, especially at high temperatures.

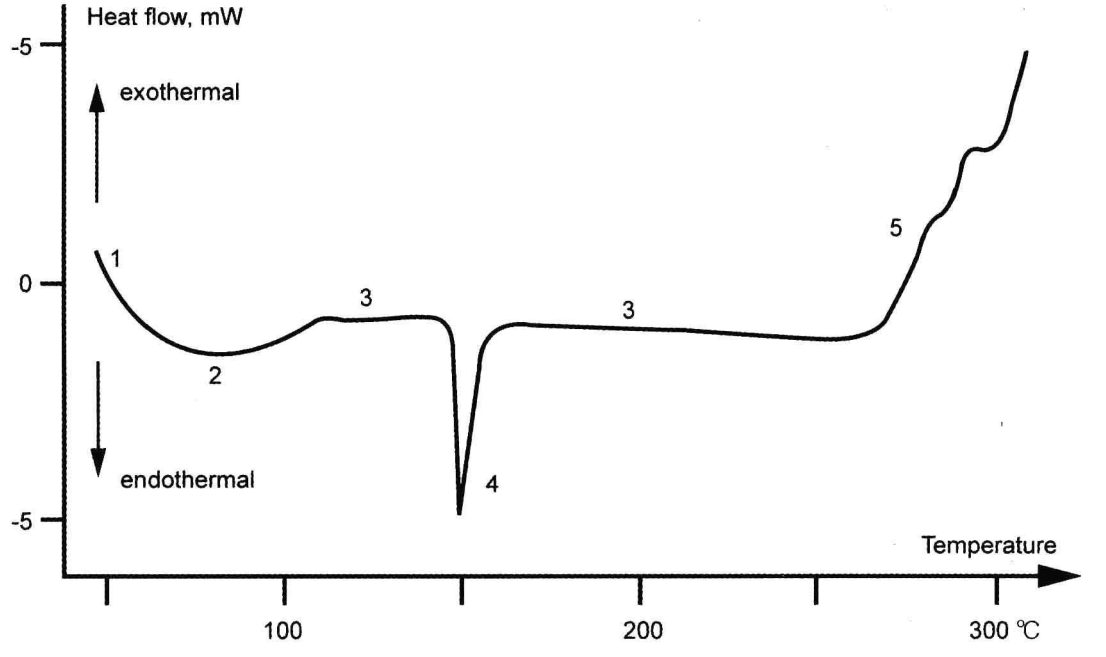


图 1.1　DSC 曲线示意图

1 初始启动偏移；2 水汽蒸发；3 基线(无热效应的 DSC 曲线部分)；4 熔融峰；5 分解开始

Figure 1.1　Schematic DSC curve

1 initial startup deflection; 2 evaporation of moisture; 3 part of DSC curve with no thermal effects, i.e., baseline; 4 melting peak; 5 beginning of decomposition.

可通过冷却和再次测试试样来区分物理转变与化学反应—化学反应是不可逆的，而熔化了的结晶物质当冷却或二次升温时会重新结晶。玻璃化转变也是可逆的，但经常在玻璃化转变的第一次升温测试中观察到的焓松弛是不可逆的。

Physical transitions and chemical reactions can be differentiated by cooling the sample and measuring it again-chemical reactions are irreversible whereas crystalline materials melt then crystallize again on cooling or on heating a second time. Glass transitions are also reversible but not the enthalpy relaxation often observed in the first heating measurement of a glass transition.

1.1.2 温度调制 DSC(MTDSC) Temperature-modulated DSC

1.1.2.1 ADSC

调制 DSC(ADSC)是一种专门的温度调制 DSC(MTDSC)。与常规 DSC 不同,在线性温度程序上叠加一个小的周期性温度变化。温度程序可以基础升温速率、温度振幅和周期性变化温度的持续时间来表征(图 1.2)。对于准等温测试,基础升温速率也可为零。

Alternating DSC (ADSC) is a particular type of temperature-modulated DSC (TMDSC). In contrast to conventional DSC, the linear temperature program is overlaid with a small periodic temperature change. The temperature program is characterized by the underlying heating rate, the temperature amplitude and the duration of the periodically changing temperature (Fig. 1.2). With quasi-isothermal measurements, the underlying heating rate can also be zero.

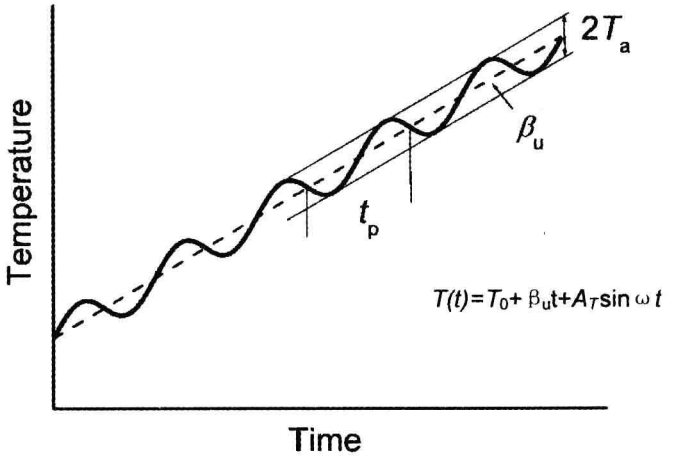
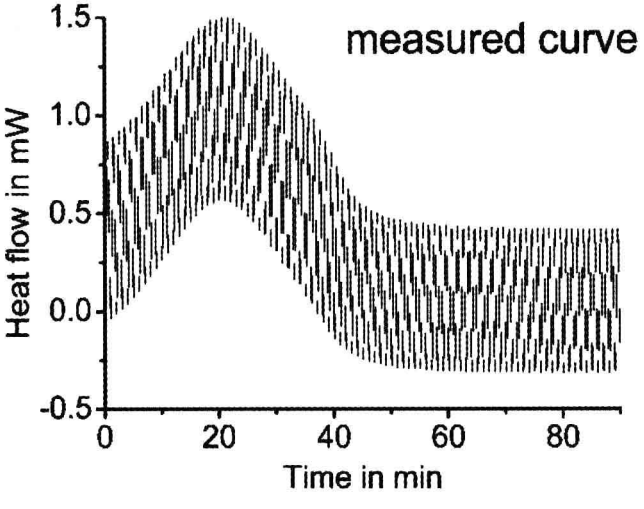


图 1.2 典型的 ADSC 温度程序

β_u 为基础升温速率,A_T 为温度振幅,t_p 为周期。$2\pi/P$ 为角频率 ω,P 为正弦波的周期

Figure 1.2 Typical ADSC temperature program

β_u is the underlying heating rate, A_T the temperature amplitude, t_p period. The angular frequency ω is defined as $2\pi/P$ where P denotes the period of the sine wave.



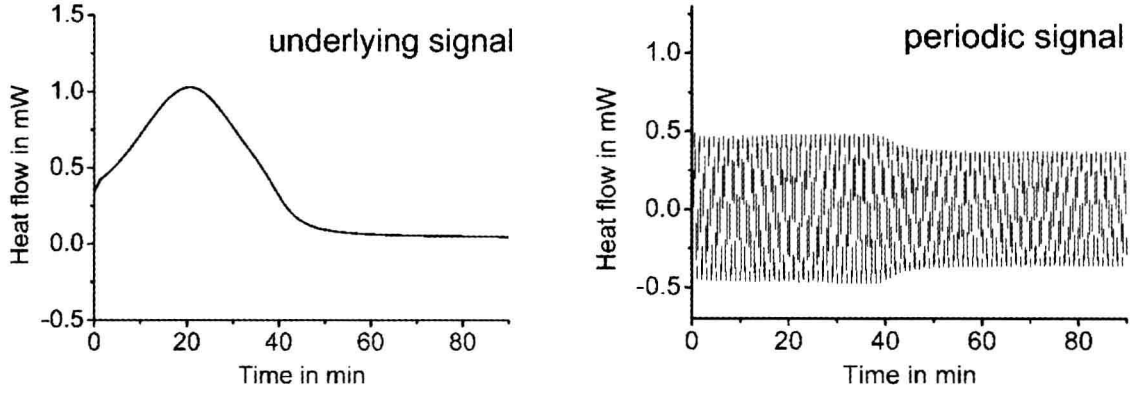


图 1.3　测得的 ADSC 曲线分离成基础和周期性信号成分

Figure 1.3　Separation of the measured ADSC curve into the underlying and the periodic signal components

温度调制的结果，是所测得的热流量周期性变化。该热流量可分成两部分，如图 1.3 所示。由信号平均得到基础信号（总热流），它相当于常规 DSC 曲线。作为附加信息，还得到周期性信号分量。可逆热流量为能够直接跟上升温速率变化的热流分量，从同相热容计算得到。总热流量减去可逆热流量得到不可逆热流量。该技术的优势之一是可将同时发生的过程分开。例如，可直接测量化学反应过程中的热容变化。

ADSC 曲线的计算以傅立叶分析为基础。复合热容 c_p^* 的复数模用下面的等式计算：

$$|c_p^*| = \frac{A_\Phi}{A_\beta} \cdot \frac{1}{m}$$

式中 A_Φ 和 A_β 为调制热流量和升温速率的振幅，m 为试样质量。ADSC 热流信号与升温速率之间的相角用于计算同相的 c_p。

As a result of temperature modulation, the measured heat flow changes periodically. This can be separated into two parts as shown in Figure 1.3. Signal averaging yields the underlying signal (total heat flow), which corresponds to the conventional DSC curve. As additional information, one also obtains the periodic signal component. The reversing heat flow corresponds to the heat flow component that is able to follow the heating rate change directly and is computed from the in-phase heat capacity. The difference between the total heat flow and the reversing heat flow yields the non-reversing heat flow. One advantage of this technique is that it allows processes that occur simultaneously to be separated. For example, the change in heat capacity during a chemical reaction can be measured directly.

The evaluation of the ADSC curves is based on Fourier analysis. The modulus of the complex heat capacity c_p^* is calculated using the equation

$$|c_p^*| = \frac{A_\Phi}{A_\beta} \cdot \frac{1}{m}$$

where A_Φ and A_β denote the amplitudes of the modulated heat flow and heating rate, and m the sample mass. The phase angle between the ADSC heat flow signal and the heating rate is used to calculate the in-phase c_p.

1.1.2.2　IsoStep

IsoStep 是一种特殊的温度调制 DSC。这种方法温度程序是由很多开始和结束为等温段的动态程序段组成（图 1.4）。

IsoStep is a special type of temperature-modulated DSC. In this method, the temperature program consists of a number of dynamic segments that begin and end with an isothermal segment (Fig. 1.4).

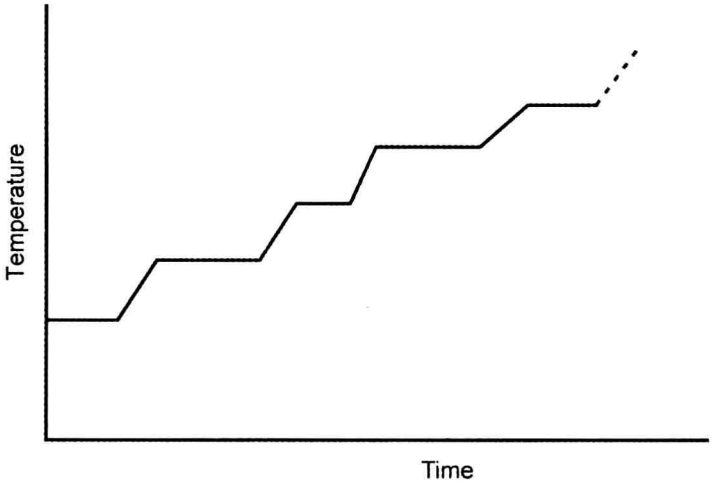


图 1.4　IsoStep 温度程序由不同的恒温和动态段组成

Fig. 1.4　IsoStep temperature program consisting of different isothermal and dynamic segments

等温段能让动态段的等温漂移获得修正，结果得到更好的热容准确性。等温步阶也可能包含动力学信息，例如化学反应。热容可用蓝宝石参比样进行测定，并可从热容变化区分开动力学效应。

The isothermal segments allow the isothermal drift of the dynamic segments to be corrected. This results in better heat capacity accuracy. The isothermal step may also contain kinetic information, for example of a chemical reaction. Heat capacity determinations can be made using a sapphire reference sample, and kinetic effects can be separated from changes in heat capacity.

1.1.2.3　TOPEM™

TOPEM™是高级温度调制 DSC 技术，基于 DSC(仪器和试样两者)对随机调制基础温度程序响应(图 1.5)的全面数学分析而设计的。由于温度脉冲是随机分布的，系统的温度振荡是在宽频范围，而不是只在某单一频率(例如 ADSC)。振荡式输入信号(升温速率)和响应信号(热流量)的相关分析能得到比常规温度调制 DSC 更多的信息，不仅能将可逆与不可逆效应分开，而且还能测量试样的准稳态热容和测定与频率有关的热容值。这能用来在一次单独的测试中就区分开频率有关的松弛效应(例如玻璃化转变)和与频率无关的效应(例如化学反应)。

TOPEM™ is an advanced temperature-modulated DSC technique that is based on the full mathematical analysis of the response of a DSC (both the apparatus and the sample) to a stochastically modulated underlying temperature program (Fig. 1.5). Due to the randomly distributed temperature pulses, the system is subjected to temperature oscillations over a wide frequency range and not just at one single frequency (e.g., ADSC). An analysis of the correlation of the oscillating input signal (heating rate) and the response signal (heat flow) provides much more information than can be obtained using conventional temperature-modulated DSC. Not only can reversing and non-reversing effects be separated, but the quasi-static heat capacity of the sample is also measured and frequency-dependent heat capacity values are determined. This can be used to distinguish between frequency-dependent relaxation effects (e.g. glass transitions) and frequency-independent effects (e.g. chemical reactions) in one single measurement.

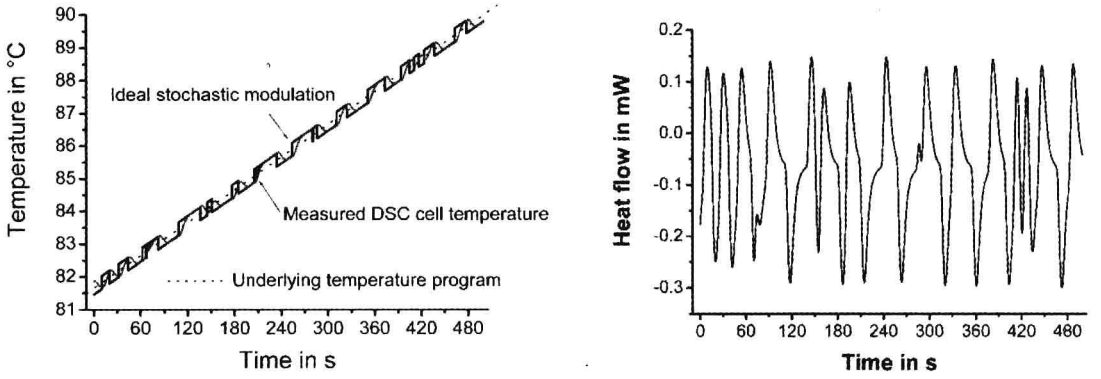


图 1.5 TOPEMTM 方法炉体设定值的温度曲线(黑线)和升温速率在平均值上下变动的炉体温度(灰线)(左图)流向试样的热流量的不规则变动(右图)

Fig 1.5 Temperature curve of the furnace set value (black line) in a TOPEMTM method in which the furnace temperature (gray curve) generates a heating rate that fluctuates around a mean value. The heat flow to the sample also fluctuates irregularly as shown in the diagram on the right.

1.2 热重分析(TGA) Thermogravimetric Analysis

当试样被加热时,经常开始失重。失重可能产生于蒸发,或形成逸出气体产物的化学反应。如果吹扫气氛是非惰性的,试样还可能与气体反应。在某些情况下,试样质量也可能增加,例如氧化反应,如果形成的产物是固体。

When a sample is heated, it often begins to lose mass. This loss of mass can result from vaporization or from a chemical reaction in which gaseous products are formed and evolved from the sample. If the purge gas atmosphere is not inert, the sample can also react with the gas. In some cases, the sample mass may also increase, e. g. in an oxidation reaction if the product formed is a solid.

热重分析(TGA)是测量试样质量随温度或时间的变化。

In thermogravimetric analysis (TGA), the change in mass of a sample is measured as a function of temperature or time.

由 TGA 可获得关于试样性质及其成分的信息。如果试样分解产生于化学反应,则试样质量通常呈台阶状变化。台阶出现时的温度可表征该样品在所用气氛中的稳定性。

TGA provides information on the properties of the sample and its composition. If the sample decomposes as a result of a chemical reaction, the mass of the sample often changes in a stepwise fashion. The temperature at which the step occurs characterizes the stability of the sample material in the atmosphere used.

典型的 TGA 曲线如图 1.6 所示。通过分析单独质量台阶的温度和高度可确定材料的组成。

Figure 1.6 shows a typical TGA curve. The composition of a material can be determined by analyzing the temperatures and the heights of the individual mass steps.

水、残留溶剂等挥发性化合物在相对低的温度逸出(曲线 1)。这些化合物的排除与气体压力有关,在低压下(真空),相应的失重台阶向低温移动,蒸发加速。通常在比挥发性化合物蒸发温度更高的温度失去结晶水(曲线 2)。

Volatile compounds such as water, residual solvents or added oils are evolved at relatively low temperatures. The elimination of such components depends on the gas pressure. At low pressures (vacuum), the corresponding mass loss step is shifted to lower temperatures, that is, vaporization is accelerated. ? Loss of water of crystallization occurs usually above the temperature at which volatile compounds evalporate (2).

更高温度,则试样的主成分发生分解,形成一个较大的台阶(曲线 3)。

At higher temperature, the main component decompose and produces a rather high step (3). The gases used can be air or

所用气氛可以是空气或氧气，也可以是氮气等惰性气体。分析在惰性气氛中的热解反应可从台阶高度确定组分含量，甚至可确定是何种物质。由残留物可测定填料或灰分。由于浮力效应和气流速率而产生的测试曲线的微小变化，可通过减去空白曲线得以修正。

oxygen, or inert gases such as nitrygen. The analysis of pyrolysis reactions in an inert atmosphere allows the content (from the step height) and possibly even the type of material to be determined.

The filler or ash is determined from the residue. Small changes in the measurement curve due to buoyancy effects and gas flow rate can be corrected by subtracting a blank curve.

TGA 测试结果经常用 TGA 曲线的一阶微商（称为 DTG 曲线）表示。于是，TGA 曲线质量损失的台阶，则在 DTG 曲线呈现峰形。DTG 曲线相当于试样质量变化的速率。

TGA measurements are often displayed as the first derivative of the TGA curve, the so-called DTG curve. Steps due to loss of mass in the TGA curve then appear as peaks in the DTG curve. The DTG curve corresponds to the rate of change of sample mass.

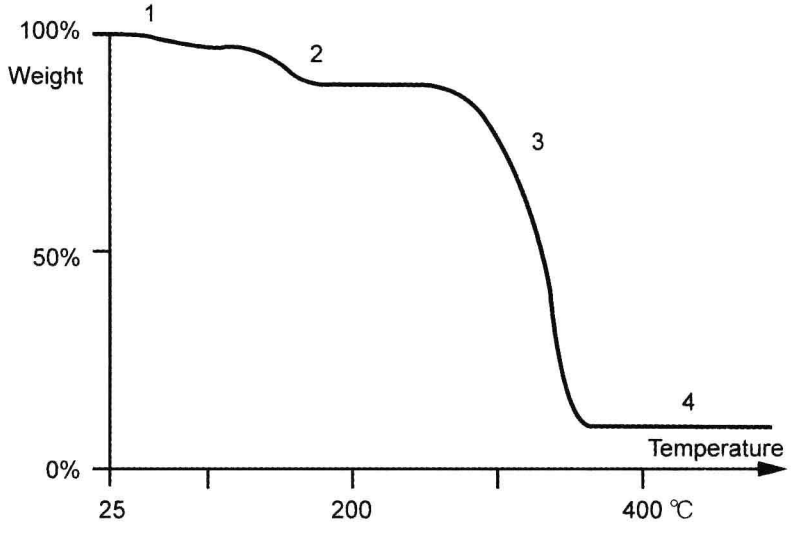


图 1.6　TGA 曲线示意图

1 挥发性成分蒸发的失重（水分、溶剂）；2 失结晶水；3 分解；4 残留物（灰分、填料、在惰性气氛中生成的炭黑或烟灰）

Figure 1.6　Schematic TGA curve

1 loss of mass due to the vaporization of volatile components (moisture, solvents); 2 loss of water of crystallization; 3 decomposition; 4 residue (ash, fillers, carbon black or soot formed during decomposition in an inert atmosphere).

分解台阶的温度范围，在一定程度上受气体产物扩散性（即从试样扩散出去的容易程度）的影响。当使用反应性气体时，试样表面气体交换的效率是关键。可以使用合适的坩埚（例如 30 μL 氧化铝等浅皿坩埚）和合适的试样几何形状（几个小颗粒或粉末）来降低测试时的扩散效应。

The temperature range of the decomposition steps is influenced to a certain extent by the ease with which the gaseous products are able to diffuse out of the sample. When reactive atmospheres are used, the efficiency of gas exchange at the surface of the sample is crucial. The effects of diffusion on the measurement can be reduced by using suitable crucibles (e. g. crucibles with low wall-heights such as the 30-μL alumina crucible) and by suitable sample geometry (several small pieces or powder).

TGA 非常精确地测量试样质量的变化。然而令人遗憾的是，该技术不提供有关逸出气体分解产物性质

In TGA, the change in mass of the sample is measured very accurately. Unfortunately, however, the technique does not provide any information about the nature of the gaseous

的任何信息。不过，通过 TGA 与合适的气体分析仪耦联(逸出气体分析 EGA)可分析这些产物。

decomposition products evolved. The products can however be analyzed by coupling the TGA to a suitable gas analyzer (evolved gas analysis, EGA).

1.3 热机械分析(TMA) Thermomechanical analysis

热机械分析测试试样升温时的尺寸变化。该项技术，连续测量带一定力放置于试样表面的探头的位置或位移与温度或时间之间的关系。图 1.7 所示为典型的 TMA 曲线。探头施加的压力和试样的硬度决定了 TMA 实验事实上是膨胀还是穿透测试。

对于热膨胀测，探头在试样表面仅施加低压力。试样在所关心的温度范围内线性升温。直接由测试曲线计算线性热膨胀系数(CTE)。

针入实验探头施加的压力大得多。可直接测量试样升温时的软化温度。物质在玻璃化转变温度处或熔融时软化。

如果对试样施加周期性变化的力，试样尺寸也周期性变化。该测试模式称为动态负载 TMA(DLTMA)。从振幅和试样厚度能估算出试样的弹性模量(杨氏模量)。

Thermomechanical analysis measures the dimensional changes of a sample as it is heated. In this technique, the position or displacement of a probe resting on the surface of the sample with a certain force is continuously measured as a function of temperature or time. Figure 1.7 shows a typical TMA curve. The pressure exerted by the probe and the hardness of the sample determine whether the TMA experiment is in fact an expansion or a penetration measurement.

In the thermal expansion measurement, the probe exerts only a low pressure on the surface of the sample. The sample is heated linearly over the temperature range of interest. The linear coefficient of thermal expansion (CTE) is calculated directly from the measurement curve.

In a penetration experiment, the probe exerts a much greater pressure. The softening temperature can be directly measured when the sample is heated. Materials soften at the glass transition temperature or on melting.

If a periodically changing force is applied to the sample, the sample dimensions also change periodically. This measurement mode is called dynamic load TMA, DLTMA. The elastic modulus (Young's modulus) of the sample can be estimated from the amplitude and the sample thickness.

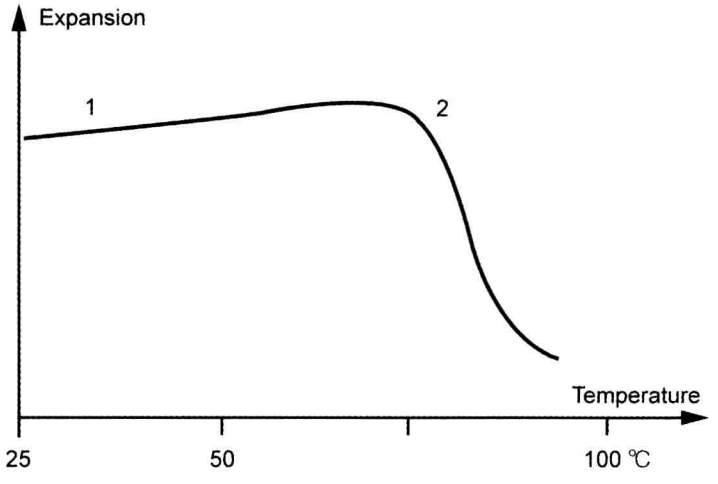


图 1.7 TMA 曲线示意图

1 低于玻璃化转变时的膨胀；2 软化(塑性形变)

Figure 1.7 Schematic TMA curve

1 expansion below the glass transition; 2 softening (plastic deformation).