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INDUSTRY FOCUS

Thermal Analysis

An exclusive collection featuring top-tier articles, visionary experts, and essential industry insights

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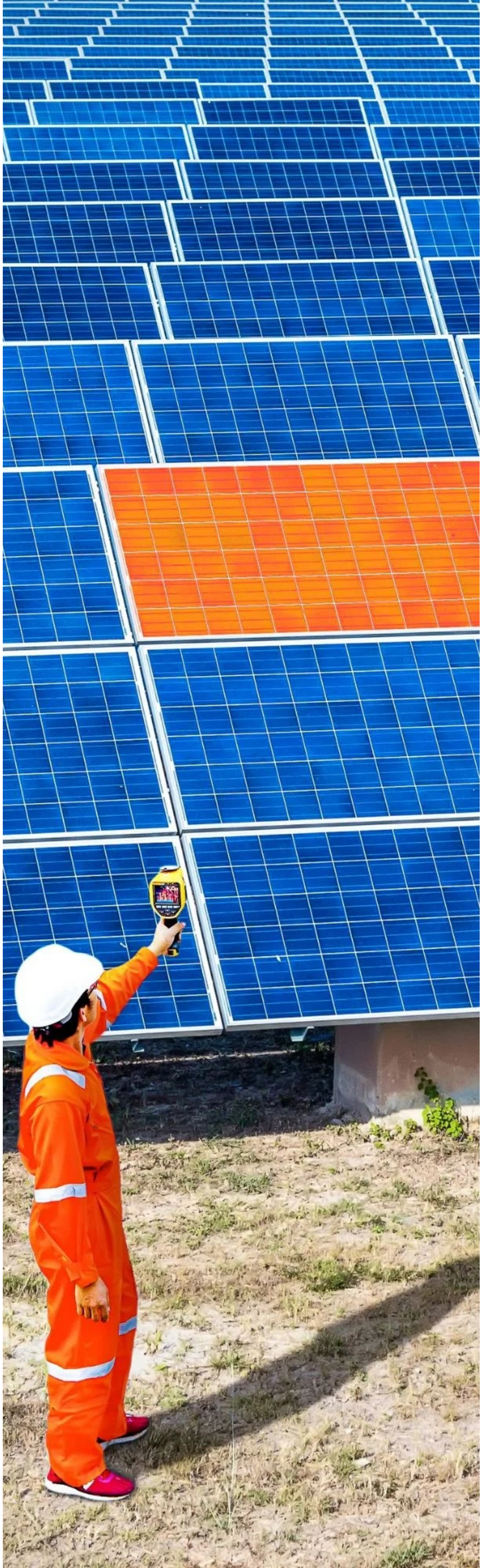

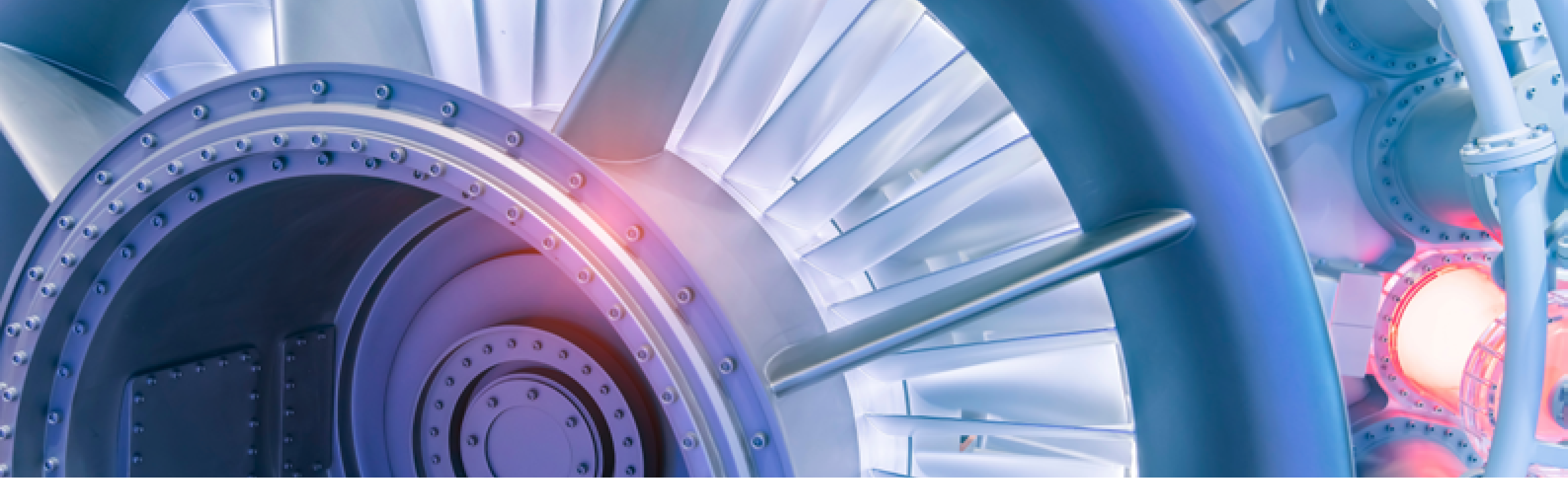

Foreword

Welcome to the latest edition of our Industry Focus eBook, where we explore the fascinating world of thermal analysis. This critical field plays a vital role in ensuring the efficiency, safety, and performance of materials and systems across aerospace and energy storage industries. As the demand for high performance materials grows, so does the importance of understanding their thermal properties and behavior.

Precise thermal analysis is essential for maintaining the integrity of astronomical instrumentation in space exploration. Our feature, **Exploring the Frontier of Space: Thermal Analysis in Astronomical Instrumentation**, delves into the techniques used to manage extreme temperature fluctuations in space.

Back on Earth, the electric vehicle (EV) industry is rapidly evolving, necessitating advanced thermal management solutions. **Thermal Management Solutions for Electronics Packaging in Electric Vehicles** examines the latest strategies to enhance battery performance and longevity in EVs.

Energy storage is another key area benefiting from thermal innovations. **Enhanced Thermal Storage Performance of Copper Foam-Phase Change Material Composites** explores how novel materials improve thermal energy storage efficiency, paving the way for more sustainable energy solutions.

Temperature-sensitive materials also play a crucial role in safety and detection systems. **Thermochromic Polymers for Heat Detection Systems** highlight how these advanced polymers are leveraged for responsive heat detection applications.

Metallurgy remains a cornerstone of industrial advancements, and understanding heat treatment effects is essential for optimizing material properties. **Microstructural Changes in Steel Under Various Heat Treatments** provides insights into how thermal processes influence steel's strength, durability, and performance.

Finally, battery technology continues to push new frontiers, with solid-state batteries promising higher energy densities and improved safety. **What Are the Latest Innovations in Solid-State Battery Technologies?** offers a closer look at the breakthroughs shaping the future of energy storage.

This eBook brings together cutting-edge research and industry insights to showcase the significance of thermal analysis in shaping modern technological advancements. We hope these articles inspire you and provide valuable knowledge as you navigate the evolving landscape of thermal management and material science.

The Role of Thermal Analysis in Pharmaceutical Testing and R&D

As a central pillar of modern society, the pharmaceutical industry bears the load of billions of lives around the world. In 2022, the global revenue of the pharmaceutical industry approximated $1.5 trillion, a figure reflected in two decades of significant growth.[1]

Pharmaceutical development is advanced by the continued search for new active pharmaceutical ingredients (APIs), including analogs, phytopharmaceuticals, and biopharmaceuticals, which have the potential to yield life-changing drugs. This enterprise is concomitant with an increasing trend toward designing new dosage forms and drug combinations, improving manufacturing processes, and exploring new indications for existing drugs.

Addressing the needs of the pharmaceutical industry, specifically the research, development and analysis of drugs, requires a sophisticated suite of techniques for fast and effective characterization and quality control. Thermal analysis is a family of techniques that measures the change of specific properties of materials as a function of temperature to elucidate their physical and chemical characteristics.[2,3] While thermal analysis comprises numerous methods, this article will focus on three key examples: differential scanning calorimetry, thermogravimetric analysis, and sorption analysis.

Pharma R&D Techniques

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DSC	TGA	Sorption Analysis
•Amorphicity/Crystallinity	•Thermal stability	•Moisture stability of API and excipient
•Influence of additives	•Shelf-life time	•Storage stability
•Polymorphs	•Composition analysis	•Detect and quantify amorphous content
•Compatibility of active ingredients and excipients	•Impurity identification	•Hygroscopicity
•Interpretation of complex systems	•Compatibility of active ingredients and excipients	•Amorphous-crystalline phase changes
•Purity	•Quality control	•Hydrate formation and dehydration
•Freeze-drying optimization		•Moisture diffusion and permeability
•Quality control		

Image Credit: TA Instruments

Discussion

Thermogravimetric Analysis (TGA)

TGA determines mass changes in a sample while it is heated or maintained under isothermal conditions within a controlled atmosphere.

A TA Instruments™ Discovery™ TGA system consists of three main components: a precision weighing system, a furnace, and a device to regulate the surrounding atmosphere. During analysis, both temperature and mass are continuously recorded. Most materials exhibit mass loss when heated; however, in some cases, such as exposure to oxygen, mass gain can occur due to oxidation.

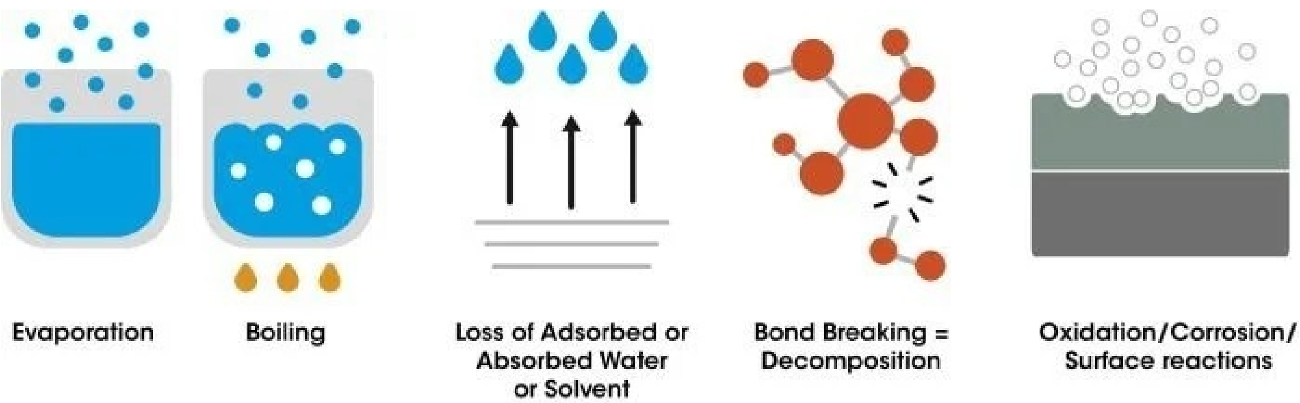

Figure 1. *Possible causes of mass loss during heating. Image Credit: TA Instruments*

A TGA analysis provides the following information:

- Loss of volatile fractions – water or solvent residues
- Thermal stability of materials
- Oxidative stability of materials
- Quantitative composition of multi-component systems
- Data on decomposition kinetics
- Anticipated operational lifespan

Figure 2. *TA Instruments Thermogravimetric analyzer (TGA). Image Credit: TA Instruments*

In a typical TGA measurement, mass loss (in %) is plotted against temperature (in °C), while the dissipation signal (shown in blue) provides additional insight into the reaction rate between different thermal events.

The peak of this derivative curve indicates the temperature at which the reaction rate is highest. Conversely, the minimum—representing the lowest reaction rate between two thermal events—is used to define the evaluation limits of consecutive decomposition stages. The curve typically begins with moisture loss, followed by multi-stage thermal decomposition.

TGA systems can also be coupled with gas analysis tools such as MS, FTIR, or GC-MS. In these setups, gases released during the experiment are carried by the purge gas to the analytical instrument, allowing for qualitative identification of volatile components and decomposition products.

Because these systems can be triggered simultaneously, data from both TGA and gas analysis can be aligned on a time scale.

Figure 4 illustrates this by overlaying the TGA curve with mass spectrometry signals corresponding to mass-to-charge ratios of 18, 36, and 43. This allows the different degradation stages to be linked to specific solvent releases.

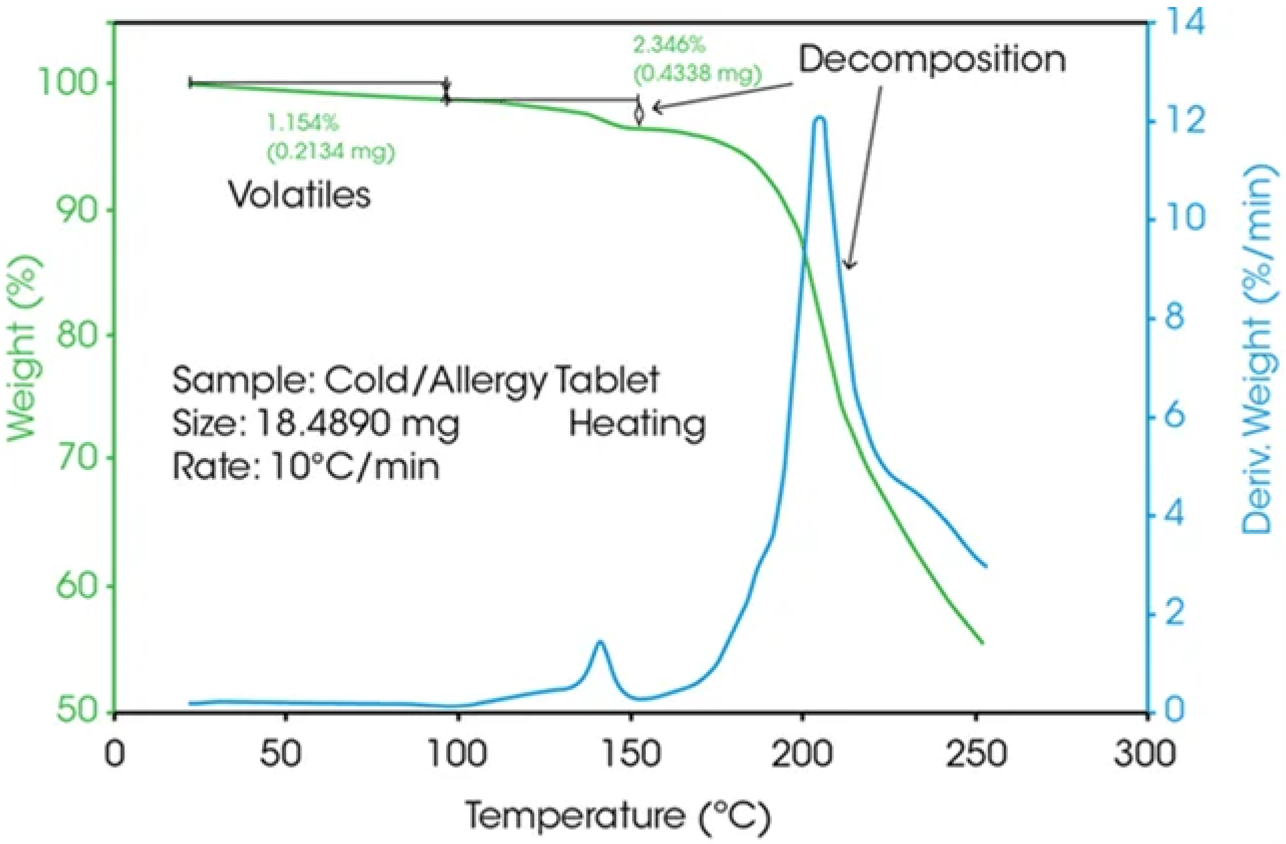

Figure 3. *TGA analysis of an allergy tablet. Image Credit: TA Instruments*

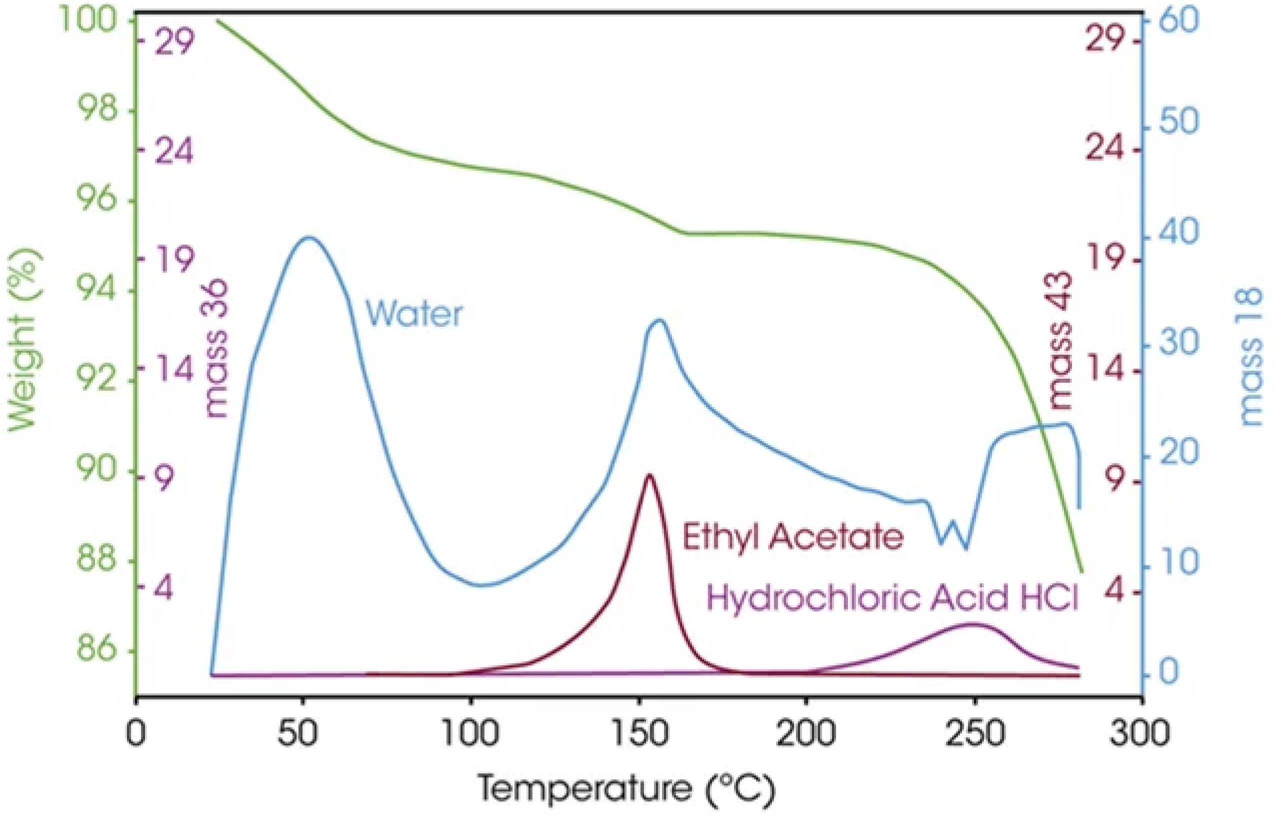

Figure 4. *TGA-MS analysis for the characterization of solvents. Image Credit: TA Instruments*

Sorption Analysis

The moisture content of solid dosage forms is a key parameter affecting stability. A Dynamic Vapor Sorption Analyzer (DVSA) is utilized to identify the change in mass under water (Steam/Relative Humidity) and can be characterized by:

- Adsorption (mass increase)
- Desorption (mass decrease)

Mass change is determined as a function of the relative humidity (RH) and the temperature (T). The TA Instruments Discovery SA instrument employs a double humidity chamber in place of the TGA furnace and can operate in the 5–85 °C temperature range. By mixing dry and moist gas streams, relative humidity can be set from 0–98 %.

This configuration supports analysis of different interactions with water:

- Wetting
- Drying
- Hygroscopy
- Hydrothermal stability

- Hydrate formations
- Moisture-induced structural changes

Figure 5. *TA Instruments Discovery SA with double humidity chamber. Image Credit: TA Instruments*

Water vapor sorption depends on the structure of the material. Chemically identical materials tend to absorb more water in an amorphous state, compared to the crystalline structure.

Water sorption can substantially decrease the glass transition temperature and trigger undesired (re-)crystallization. The physical properties of the amorphous and crystalline phases differ significantly and can impact various product attributes:

- Mechanical (pouring behavior)
- Physical (solubility)
- Chemical (stability)
- Pharmacological (bioavailability)

Sorption analysis is utilized to identify the relative humidity and temperature settings at which a material crystallizes. The amorphous lactose sample presented in Figure 6 absorbs more water at 25 °C above the glass transition, resulting in recrystallization. This information is critical for determining appropriate storage and transport conditions.

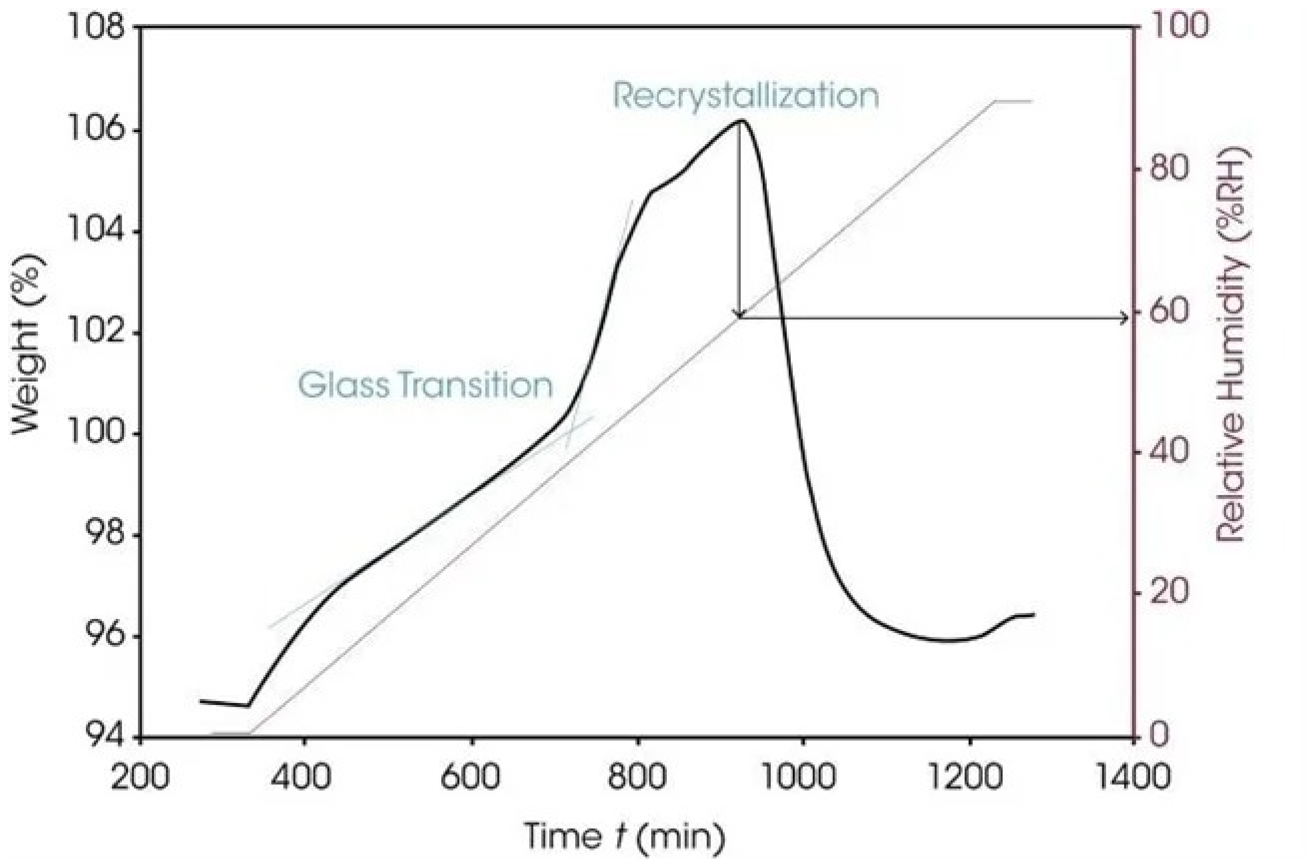

Figure 6. *Water sorption of amorphous lactose. Image Credit: TA Instruments*

Differential Scanning Calorimetry (DSC)

DSC determines the heat flux that is released or absorbed as a function of time, temperature, and atmosphere. It is always measured against a reference sample, typically an empty pan. Open, closed, or hermetically sealed pans are utilized as sample vessels, with aluminum pans being most frequently utilized.

Since measurements can be conducted in heating, cooling, or isotherm, DSC systems are usually combined with a cooler (compressor cooler or liquid nitrogen).

The heat flow reveals:

- Phases or structural alterations
- Chemical reactions and physical interactions

DSC can be utilized to identify material parameters, such as:

- Melting temperature
- Melting enthalpy
- Glass transition temperature
- Influence of cooling rate and storage on the formation of the amorphous and crystalline phases
- Purity determination
- Compatibility
- Polymorphism
- Denaturation

Figure 7. *DSC cell. Image Credit: TA Instruments*

Compatibility

Component incompatibility can impact product longevity or efficacy. Determining the individual components in comparison to a mixture of the components can provide information about this.

Magnesium stearate, commonly utilized as a lubricant in pharmaceutical tablet fabrication, is presented as an example of compatibility testing using TA Instruments Discovery DSC in

Figures 8 and 9. Figure 8 compares the 50/50 mixture of aspirin and magnesium stearate with the pure materials. Each of the individual components exhibits an endothermic melting peak.

A compatible mixture would display two melting peaks of proportional size in the same temperature window. However, the mixture reveals two endothermic effects at substantially lower temperatures, clearly indicating incompatibility.

To evaluate material compatibility at room temperature or below their melting points, material heat capacity can be determined as a single substance and, in comparison, as a mixture instead of the heat flow. Modulated DSC™ (MDSC™) experiments provide significant benefits here.

This method, which does not rely on sinusoidal temperature oscillation, allows for the isothermal determination of heat capacity (Cp). Figure 9 shows the stable Cp curves of each substance, recorded over time at a constant temperature of 50 °C.

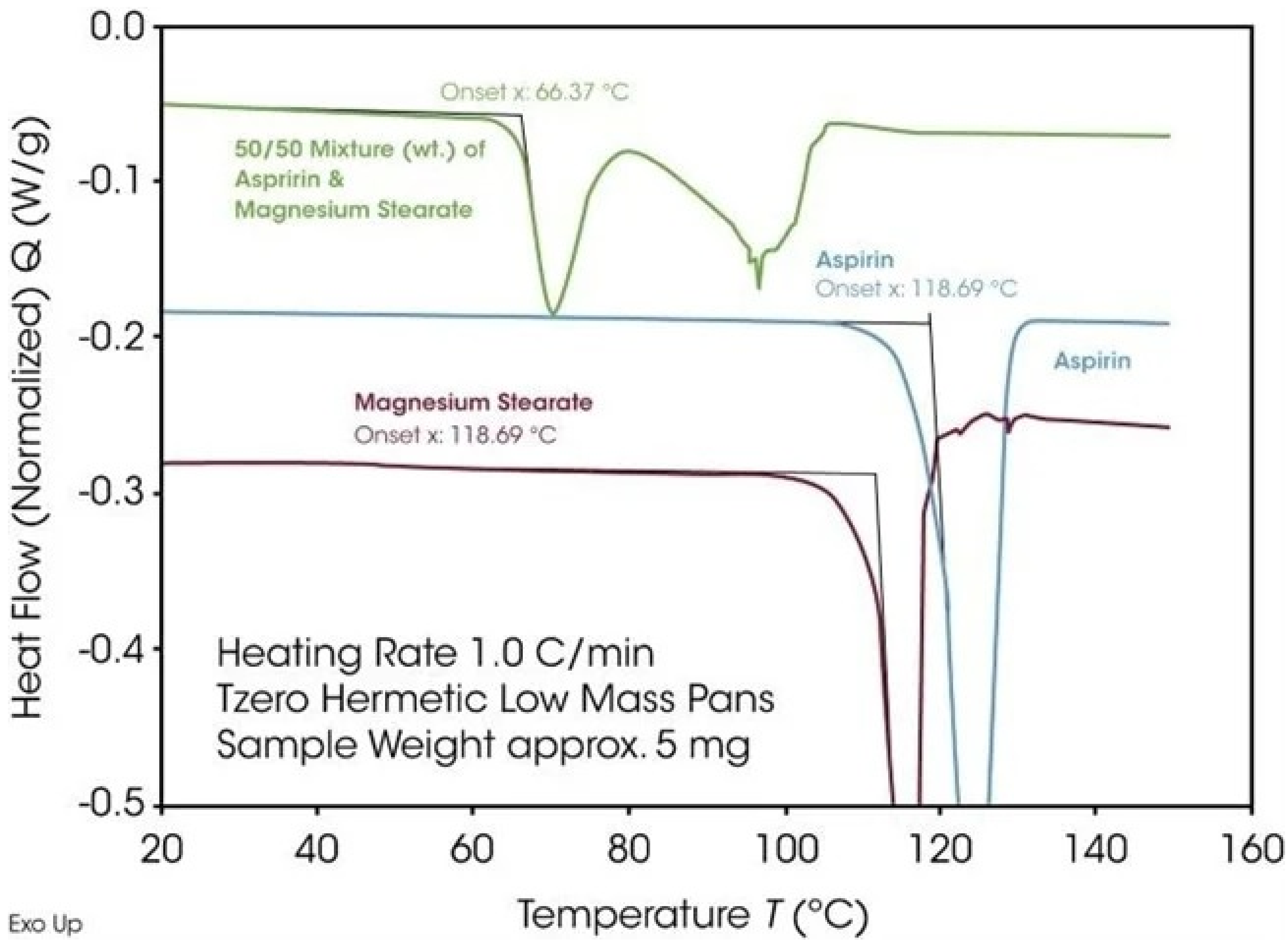

Figure 8. *Compatibility check using DSC. Image Credit: TA Instruments*

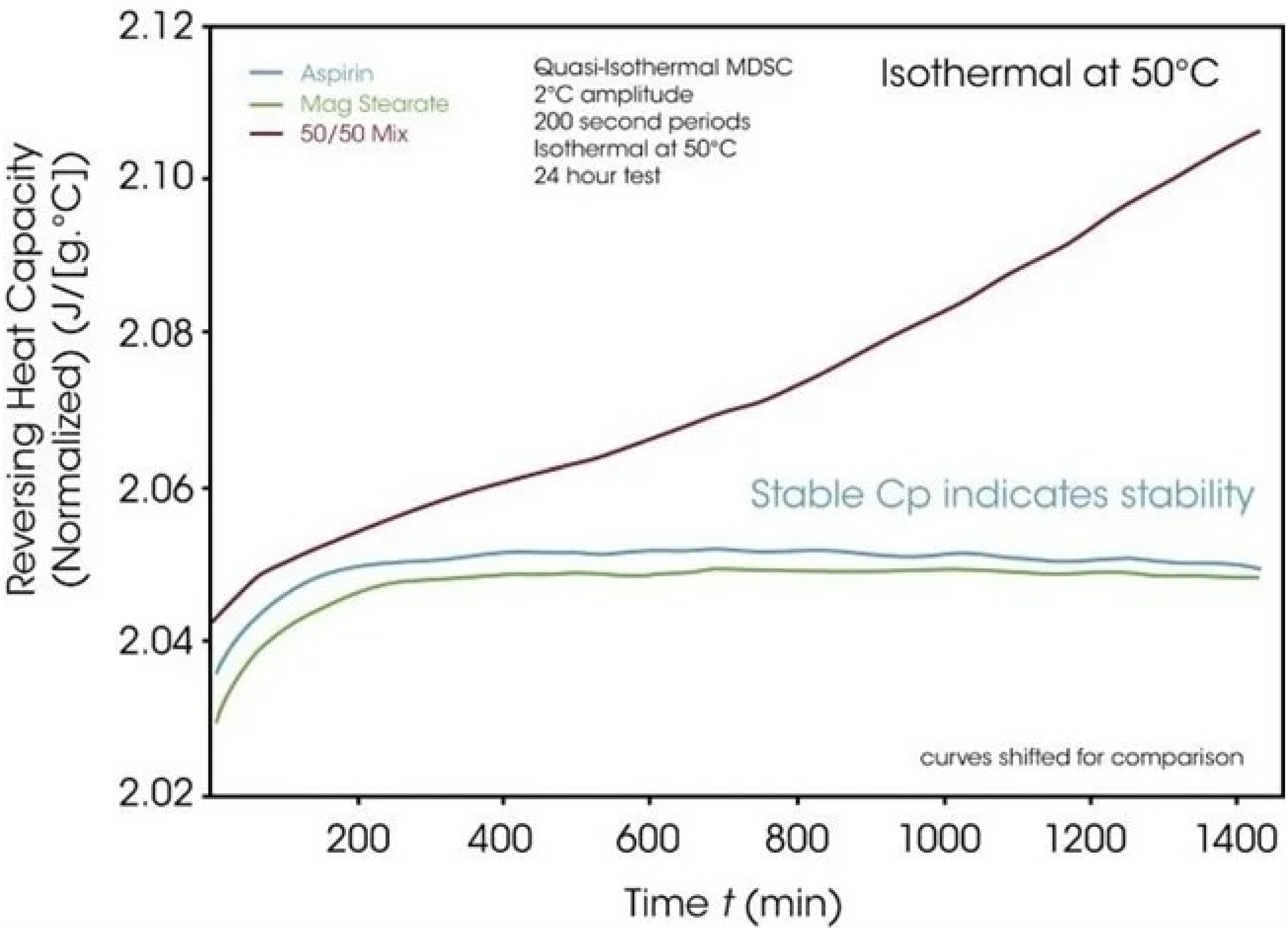

Figure 9. *Quasi-isothermal Cp determination to test compatibility. Image Credit: TA Instruments*

Glass Transition

An important aspect of pharmaceutical material analysis is identifying the glass transition using DSC.

The proportion of the amorphous phase in an active pharmaceutical ingredient (API) or finished product significantly affects characteristics like tabletability, solubility, and, ultimately, the drug's release rate and bioavailability. However, amorphous and semi-amorphous substances are thermodynamically unstable and prone to recrystallization.

Humidity can lower the glass transition temperature (Tg) below ambient conditions, potentially triggering crystallization. This may result in the formation of polymorphic forms or hydrates, as well as particle agglomeration.

Moisture uptake can also impact the physical properties of the dosage form. In a DSC measurement, the glass transition appears as a step-like change in the heat flow signal. Both the onset temperature and the height of this step are critical, as shifts in these values can reveal information about the material's amorphous content.

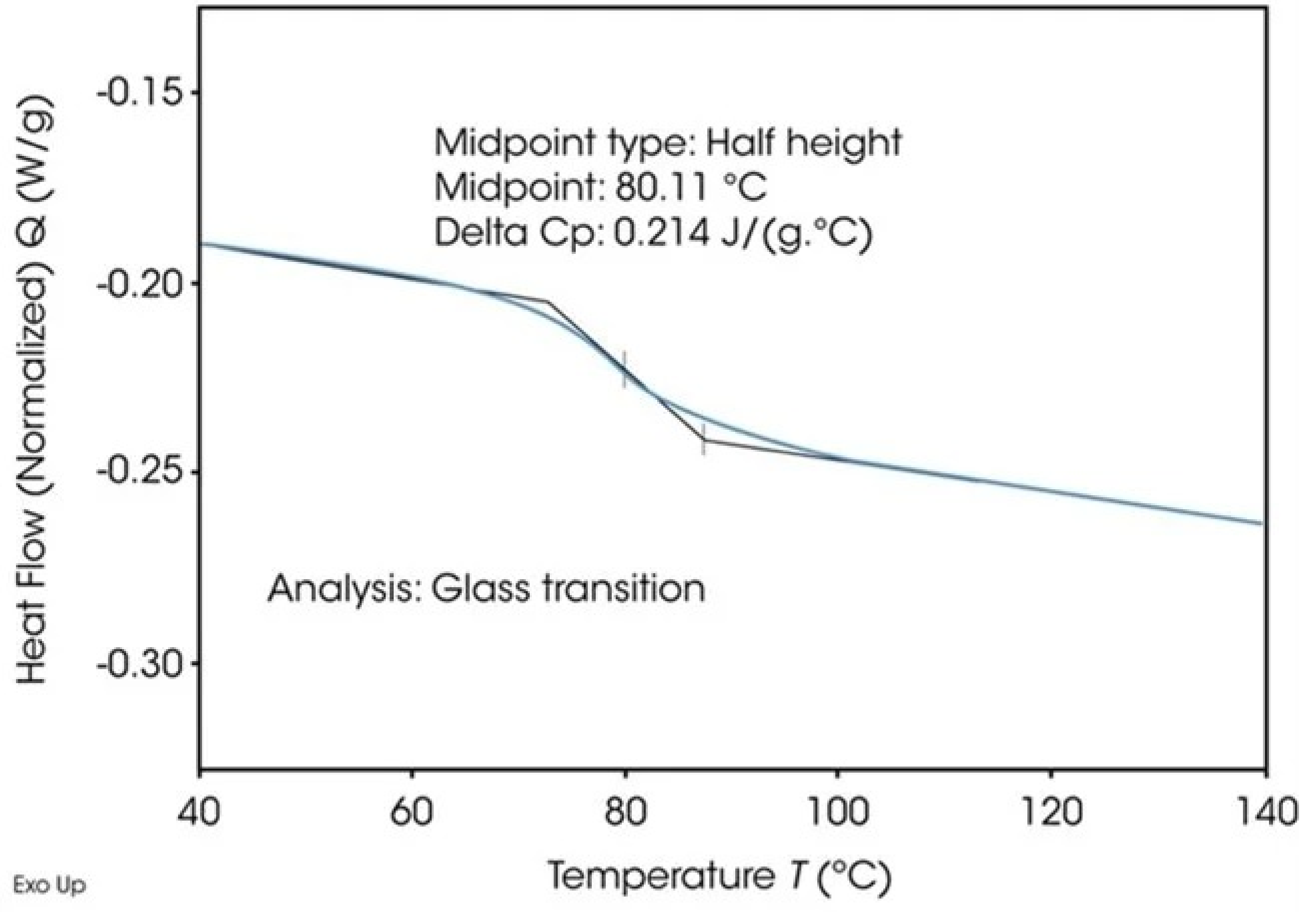

Figure 10. *Typical glass transition determined by DSC. Image Credit: TA Instruments*

Summary

Thermal analysis techniques such as TGA, SA, and DSC provide powerful tools for evaluating the material properties of active pharmaceutical ingredients, carrier substances, and finished products. These methods are especially advantageous in research settings where only small sample quantities are available, as they require minimal material for testing.

Compared to many other analytical approaches, DSC and TGA offer relatively fast analysis times, allowing researchers to obtain meaningful data within short measurement intervals.

Acknowledgments

Produced from materials originally authored by Monika Schennen.

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Exploring the Frontier of Space: Thermal Analysis in Astronomical Instrumentation

Astronomy has always relied on optical instruments, from the early Galilean telescope to fiber-fed spectrographs, so much so that astronomy and modern optics are called astrophotonics. Since astronomical observations depend on electromagnetic radiation, optical instruments are critical for advancing our understanding of the universe.

Image Credit: Dima Zel/Shutterstock.com

Astronomical instruments must operate efficiently across a wide range of daytime or night-time temperatures.[1] Solving complex astronomical problems requires instruments with extreme characteristics, highly dependent on temperature.[2] Thermal analysis is, therefore, pivotal for optimizing these instruments, ensuring precision in extreme conditions.[1]

Science of Thermal Optical Analysis

Temperature variations influence the functioning of optical systems due to thermoelastic (changes in the position and dimensions of optical elements) and thermo-optic (changes in the refractive index of optical materials) effects.

Thermoelastic effects in optical components result from the expansion and contraction of materials due to thermal strains induced by temperature variations. These appear as changes in the optical element's thickness, diameter, radii of curvature, and surface deformations.[1]

Due to the thermo-optic effect, as light travels through an optical component at varying temperatures, its path length changes, resulting in optical path differences over the aperture and giving rise to transmissive optics.

Thus, thermal optical analysis, including both thermoelastic and thermos-optic analyses, becomes crucial in optical engineering. Integrated optomechanical analysis involves representing both thermoelastic and thermo-optic errors in an optical model to predict the device's performance under complex thermal loads.[1]

All modes of heat transfer (conduction, convection, and radiation) are considered for the analysis of precision optical systems.

Radiation is the most critical aspect in astronomical applications where no air is present for convection. Convection analysis is equally important for ground-based observatories.

Even minor temperature variations can generate significant internal stress in an optical system and degrade its optical performance. Hence, thermal optical analysis is a critical aspect of astronomical instruments being developed to explore the frontier of space.[1]

Materials and Design for Thermal Stability

Glass ceramics like Zerodur (a Li_2O-Al_2O_3-SiO_2 composite) are used as mirror substrates for astronomical telescopes due to their extremely low thermal expansion coefficient, which makes them highly stable against temperature changes. The high reproducibility and homogeneity of such materials also make them suitable for application in large astronomical telescopes.[1]

In the case of telescopes operating in the ultraviolet-visible spectrum range, high-temperature gradients can cause the mirror to deform thermally. Thus, materials with ultra-low thermal expansion coefficients are required for their fabrication. For example, the mirror with a diameter of 2.4 m for the Hubble telescope is made of specially designed glass with a near-zero thermal expansion coefficient.[2]

Passive and active thermal control techniques are employed to maintain temperatures within operational limits, ensuring satisfactory performance and structural integrity. A thermal control system provides cooling, heating, insulation, or shading to ensure the system optics remain within the operational temperature range.[1]

A system for providing thermal mode (SPTM) is often used to thermally stabilize radiation detectors in astronomical instruments. It comprises both active and passive levels.

The SPTM passive level reduces temperature oscillations due to solar radiation, while the active level contains a heater and a cooler, which smooths the remaining temperature oscillations of the detector in the cold and warm orbits, respectively.[2]

Case Studies: Overcoming Thermal Challenges in Optical Systems

One of the most complex cryogenic tests executed by NASA was the Optical Telescope Element and Integrated Science Instrument Module (OTIS) Cryo-Vacuum (CV) test for the James Webb Space Telescope (JWST).

Due to its size, it was not possible to test the whole JWST observatory at a single facility. Therefore, the system-level testing (for payload, ground support equipment, and surrounding test chamber) was correlated using thermal models to achieve observatory-level verification.

The simulations, involving cooling down and warming up in a flight-like environment, were able to predict payload performance at cryo-stable conditions.[3] The successful operation of JWST with unprecedented capabilities demonstrates the efficacy of such a complex optical thermal analysis.

A structural-thermal-optical performance (STOP) analysis was recently used for the subsystems of the under-construction European Extremely Large Telescope, which will be the world's largest optical or near-infrared telescope once completed.

The team used an indigenously developed software called Sensitizer to carry out optical thermal analysis at different stages of the telescope, such as phasing and diagnostic station and pre-focal station.[4]

Thermal optical analysis is a crucial aspect of giant projects involving tremendous efforts and resources.

Innovations in Optical Thermal Analysis

Along with the physical influence of temperature on astronomical instruments, experimental and mathematical modeling of their thermal systems also presents challenges, such as accurately replicating external thermal factors for precise modeling of the instruments' thermal modes. Innovations are therefore required to increase the efficiency of modeling techniques in optical thermal analysis.

Since the different components of an instrument are manufactured in various countries, it becomes necessary to accommodate the thermal modes of each component into a single model.[2]

Thermal models have been developed to design and predict temperature distribution over optical systems. These models can be used for thermoelastic and thermos-optic analyses. Computational fluid dynamics, finite-difference methods, and finite elemental modeling are some of the commonly used optical thermal analysis techniques.

The greater the target accuracy of the system, the more intensive the computational analysis needs to be.[1] Thermal models, such as the JWST OTIS CV test, help with test and runtime scheduling and ensure hardware safety.

It was a comprehensive test of the integrated telescope system for its optical, structural, and thermal performance. Beyond optical system design and testing, these models can accurately predict the temperatures that can be expected during the flight, thereby facilitating the performance prediction of the instrument.[3]

The Future of Astronomical Exploration Instrumentation

Astronomical instruments working in different spectrum ranges encounter various challenges. For instance, microwave and infrared telescopes require their temperature to be kept close to absolute zero.

However, current cryogenic technologies have not yet achieved such low temperatures for large mirrors used in telescopes.[2] Thus, continued investment in materials research is essential for the next generation of optical technologies.

For complex systems like astronomical instruments that demand high performance in extreme environments, integrated STOP analysis is required as a thermal management strategy.[1]

Thermal management in optical systems could be revolutionized through potential breakthroughs in material science, such as artificial intelligence that facilitates the design and simulation of novel materials with targeted properties and predicts their performance in operational environments.

Such advancements will enhance the performance and durability of astronomical instruments and expand human understanding of the universe.

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Thermal Management Solutions for Electronics Packaging in Electric Vehicles

Electronic Vehicles (EVs) have been at the center of attention in recent years. With the automotive industry becoming more aligned with sustainable development goals, the focus has shifted toward EVs and improving their performance.

Image Credit: Roman Zaiets/ShutterStock

Maintaining the temperature and heat level of electronic components, batteries, and packaging materials in electric cars is a complex process. The focus for improved thermal performance initially involved researching battery cells that are safer, more durable, and capable of withstanding thermal exposure.

Subsequently, the design of battery packing has gained significance in preventing thermal events that could lead to explosions or, at the very least, mitigate potential severe consequences if an explosion occurs. An effective battery packing design aims to keep the temperature of the battery cells within the optimal range.

The Complexities of EV Electronics Packaging

Packaging companies encounter distinctive challenges when handling EVs—the delicate nature of electronic components utilized in EVs demands specialized packaging solutions. The distinct shape and size of EV parts necessitate tailor-made packaging designs to securely accommodate them.

The battery packs are generally much larger than the ones present in conventional vehicles, requiring adequate packaging to protect them from damage during travel. The rising demand for EVs also poses challenges concerning volume and scalability in the packaging industry.

The packaging material and design adopted by the companies are finalized, with considerations for the size, weight, and unique shape of the electric car's battery. Instead of using conventional materials and simple designs, modern manufacturers use specialized types of packaging for the electronics of these novel automobiles. The packaging consists of thermally insulated foam, specialized custom containers, and additional supplementary cushioning for protection against excessive force.

To reduce the carbon footprint and emission of harmful gases, packaging companies are focusing on developing strategies that minimize waste products and reduce carbon dioxide (CO_2) production. This is evident by the use of biodegradable packaging material and the optimization of conventional manufacturing processes.

How Does Battery Thermal Management System Improve Thermal Performance?

Lithium-ion batteries (LIBs) are extensively used to power modern electric cars. When an EV operates, the temperatures of the electronic components and batteries increase, requiring careful monitoring and maintenance. This is facilitated by a specialized system called the Battery Thermal Management System (BTMS).

An article published in *Batteries* highlights that BTMS designs and strategies can be classified based on their working principles and the coolant material employed for heat transport. The working principle of BTMS can involve either direct transfer between the coolant and batteries or an indirect cooling system incorporated within the BTMS.

Indirect cooling is achieved by using a pipe through which heat is released. EVs also use different types of coolant materials to ensure optimum operational temperature. BTMSs may be air-cooled systems, liquid-cooled, or use phase-change materials (PCMs) as a cooling material.

Compared to other thermal management systems, a BTMS using air as a coolant is relatively simple in design and cost-effective. However, the low heat capacity of air becomes a major limitation of these systems. In contrast, a liquid-cooled BTMS utilizes a liquid with higher heat capacity than air. As an alternative, PCMs, particularly those based on paraffin, have been developed to enhance heat capacity and are widely recommended for cooling electric vehicle batteries.

BTMS based on PCMs exhibits high efficiency and stable performance, especially under extreme conditions. This is attributed to the PCM's capacity to store heat during the phase change process. However, it is important to note that PCM materials typically have low heat conductivity. The PCM must also undergo a regeneration process after being fully melted to maintain its effectiveness.

Thermal Control of EV Power Electronics: Impact of Additive Manufacturing

The thermal management solution's design influences the reliability and power density of power electronics (PEs). In the evolving landscape of the EV industry, which strives for increased efficiency and output power, the cooling system must efficiently handle the excess heat generated in PEs.

According to an article published in *IEEE Transactions on Transportation Electrification*, indirect, direct, and double-sided cooling methods are prevalent in EVs, constituting 14 % to 33 % of the total volume of traction inverters. However, as the packaging sizes of PEs are anticipated to decrease, the challenge of dissipating increasing heat persists. Ongoing research is therefore focused on exploring advanced cooling technologies.

Power semiconductor devices serve as pivotal components in the PE systems of EVs. Elevated temperatures can induce undesirable alterations in material properties, and the mechanical stresses arising from high transient temperatures or thermal cycling may lead to mechanical failures or fatigue. Selecting suitable packaging materials plays a crucial role in preventing these failures.

Breakthroughs in the domain of additive manufacturing have enabled the companies to redesign the heat sinks, leading to significant improvements in the thermal performance of EVs. The automobile manufacturer Toyota has used additive manufacturing to develop fins made using 3D printing, resulting in improved outward heat flow.

Advanced cooling techniques, like air jet impingement, have additionally been incorporated. Additive manufacturing has also been applied to produce microchannel heat sinks to achieve superior thermal performance.

Polyurethane (PU) Foam Packaging: Enhancing EV Electronics

Polyurethane (PU) foam has recently been used in packaging electronic components of EVs. The lightweight nature of the foam packaging is a significant advantage, as the EV batteries are already heavy. Thus, using PU foam leads to considerable weight savings compared to traditional packaging materials.

PU foam has been used in various studies, demonstrating superior thermal attributes. It enables an efficient heat flow from the source to the sink, ensuring an optimum temperature is maintained when the vehicle operates. When a stable and optimum operational temperature is sustained, it minimizes the damage caused to the batteries and improves the durability of electronic components. Moisture cannot pass through this packaging, ensuring the components are safe and working correctly.

Novel Material Barriers and Packaging for Thermal Runaway Management

LIB thermal runaways can arise from mechanical and thermal stresses, potentially leading to severe collateral damage, fires, or explosions. To address this issue, a novel approach (published in the *International Journal of Heat and Mass Transfer*) involves using a "smart" nonwoven electrospun separator packaging with thermal-triggered flame-retardant properties for LIBs.

Triphenyl phosphate (TPP) is added to the separator, and during a thermal runaway event, the protective polymer shell melts due to increased temperatures. This melting triggers the release of the flame-retardant TPP, effectively suppressing the combustion of highly flammable electrolytes.

Another innovative solution involves incorporating a fast and reversible thermo-responsive polymer switching material inside batteries to prevent thermal runaway. This material comprises electrochemically stable graphene-coated spiky nickel nanoparticles mixed in a polymer matrix with a high thermal expansion coefficient. Batteries equipped with this self-regulating material within the electrode can swiftly shut down under abnormal conditions, such as overheating and short circuits. Importantly, these batteries can resume their normal function once the abnormal conditions are addressed, without compromising performance.

The packaging of electronic components and batteries in EVs plays a crucial role in ensuring the safety and durability of the components. Advances in materials science and the use of interdisciplinary technologies, such as artificial intelligence, are ensuring improved thermal management systems and better packaging designs.

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Using SDT to Analyze the Thermal Properties of LFP Li-Ion Cathodes

Lithium-ion batteries power everything from our portable electronics and electric vehicles to vital medical equipment and renewable energy storage. As the market expands, researchers are finding ways to make Li-ion batteries increasingly powerful, dependable, and safe, all while minimizing production time and cost.

Since its development in the 1990s, Lithium Iron Phosphate (LFP) has become a popular cathode material for lithium-ion batteries (LIB). Among its advantages, LFP is cost-effective, long lasting, and is one of the safest cathode material options available. It offers great thermal stability and excellent electrochemical properties, but its low electrical conductivity has led to ongoing efforts to optimize its performance.

One of the strategies used is to modify the surface of the LFP through a carbon (C) coating; however, the source of carbon can influence performance, as well as coating thickness. If the coating is too thick, it can hinder diffusion of lithium ions and decrease the energy density of the battery. As a result, an LFP/C composite material with optimized carbon source and loading is desired for high performance.[1] Other strategies include modifying LFP morphology or doping the material to improve electrical conductivity. Understanding the phase transition temperature and heat flow properties can help optimize the process, while also gaining insight into the effects of any material modifications. Oxidation can readily occur in LFPs and care needs to be taken in the process to avoid this.[2, 3]

Formulators and manufacturers need efficient means to verify the carbon content and integrity of their coatings, check for oxidation conditions, and understand the phase transition behavior. Differential scanning calorimetry (DSC) can be coupled with thermogravimetric analysis (TGA) to evaluate coated LFP.

Differential Scanning Calorimeters (DSC) measure the heat absorbed or released when a sample material is heated, cooled, or held isothermal. The heat flow is determined by comparing the heat flow difference between a sample material and a reference. DSC provides insights into the battery material's heat capacity and phase transitions such as melting point (Tm), heat of fusion, and glass transition (Tg).

Thermogravimetric Analyzers (TGA) programmatically heat up a material while measuring its mass change with a highly sensitive analytical balance. Loss of mass indicates possible decomposition or vaporization, while a gain in mass represents possible sorption or that the material is reacting with its gaseous surroundings. Battery developers turn to TGA to quantify oxidation, thermal degradation, and thermal stability. TGA elucidates the temperatures at which battery materials start to degrade, empowering researchers to choose proper materials and build high performing, long-lasting batteries.

Simultaneous **DSC-TGA** (SDT) measures both weight changes and heat flow in a material as a function of temperature or time in a controlled atmosphere up to 1500 °C. In this paper, SDT will be used to identify the composition of carbon coating on LFP from the weight change, determine the phase transition temperature, and the enthalpy of reaction during phase transitions from the heat flow data. X-ray diffraction (XRD) will be done to further investigate LFP oxidation and crystalline structure.

Experiment

Commercially sourced carbon-coated LFP powder was provided by NEI Corporation. For comparison, uncoated reference LFP was obtained from Sigma-Aldrich to evaluate coating degradation and phase transitions.

Weight loss and heat flow data for both materials were collected under nitrogen using a TA Instruments SDT 650 (Figure 1). To assess the effect of different atmospheric conditions, the coated NEI samples were also tested under argon and air purge gases.

Samples were heated from room temperature to 1200 °C at a rate of 20 °C/minute. Both alumina and sapphire pans were used, with sapphire pans recommended for experiments above the melting point to prevent adhesion to the SDT beam.

To complement the SDT results, XRD was performed to analyze changes in the crystalline structure of coated LFP after high-temperature exposure. Two annealing experiments were carried out under different environmental conditions.

In the first experiment, LFP powders were annealed in a muffle furnace by heating to 350 °C at a rate of 5 °C/minute, maintained at that temperature for two hours, then cooled at a rate of 10 °C/minute.

The second set of powders were annealed in a nitrogen atmosphere by placing the powders in a tube furnace. This tube furnace was pre-flowed with nitrogen gas for 30 min at a rate of 200 mL/min to eliminate any residual air. The powders were then heated to 950 °C at a rate of 5 °C/minute, kept at temperature for two hours, and cooled to room temperature at a rate of 10 °C/minute.

Both pristine and annealed samples were analyzed by NEI Corporation using a MiniFlex II XRD system from Rigaku Corporation to evaluate structural stability.

Figure 1. *TA Instruments SDT for simultaneous DSC and TGA measurements at high temperatures. Image Credit: TA Instruments*

Results and Discussion

Figure 2 illustrates the coating content of the commercial LFP cathode samples in nitrogen.

Coated LFP cathode (blue) exhibited a 3 % weight loss, while the uncoated LFP (green) showed negligible weight loss. Consequently, the coated LFP cathode material (blue) loses 3 wt% of the organic coating, with the remaining 97 % of the material constituting LFP.

Both LFP samples displayed an endothermic peak melting transition near 970 °C.[4] The coated LFP cathode exhibited both coating decomposition and LFP phase transition across the same temperature range. For improved precision in enthalpy values, the endothermic heat flow was plotted as weight-corrected heat flow to obtain the weight-adjusted enthalpy value.

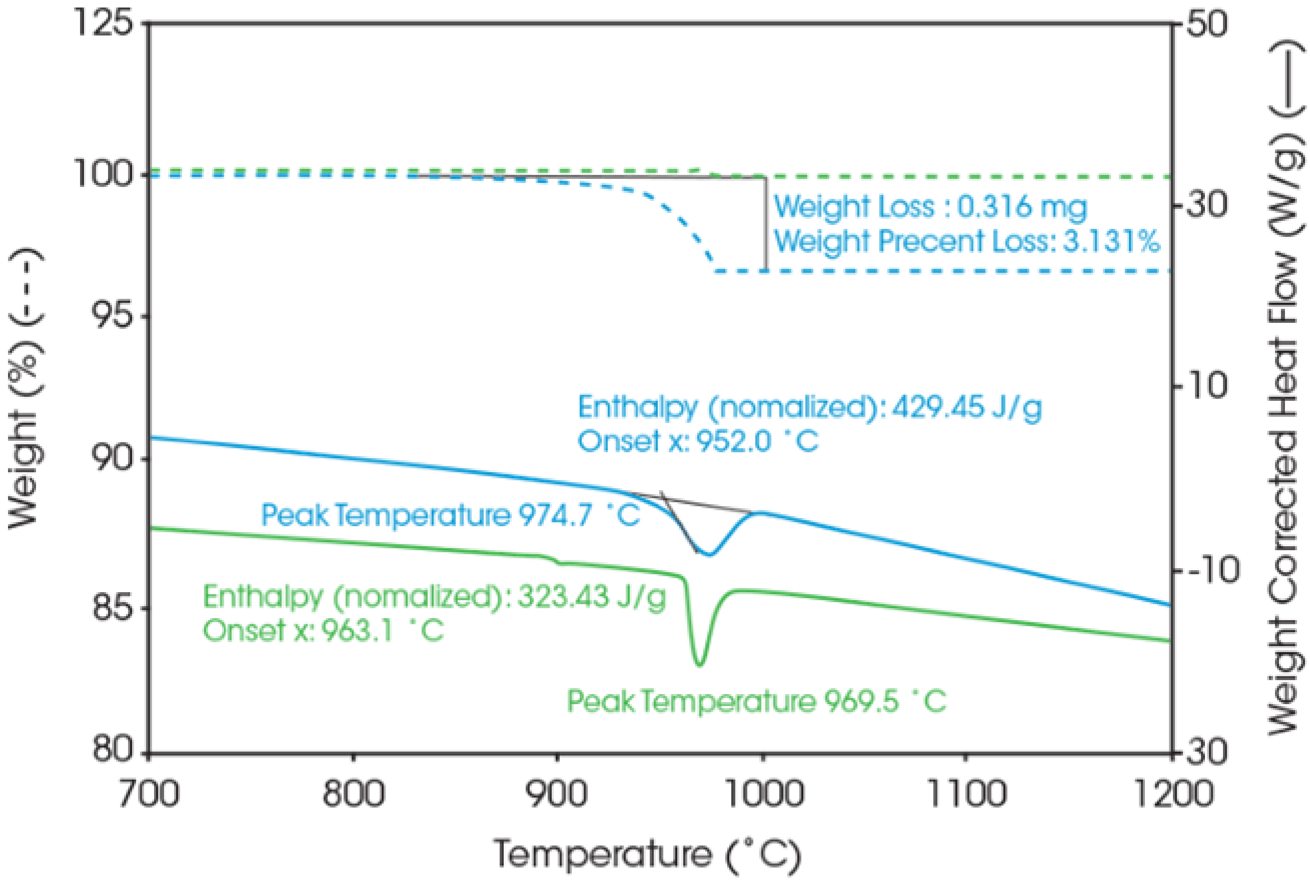

Figure 2. Weight change and heat flow of LFP samples from coated (Blue) and reference uncoated (Green) under nitrogen. Image Credit: TA Instruments

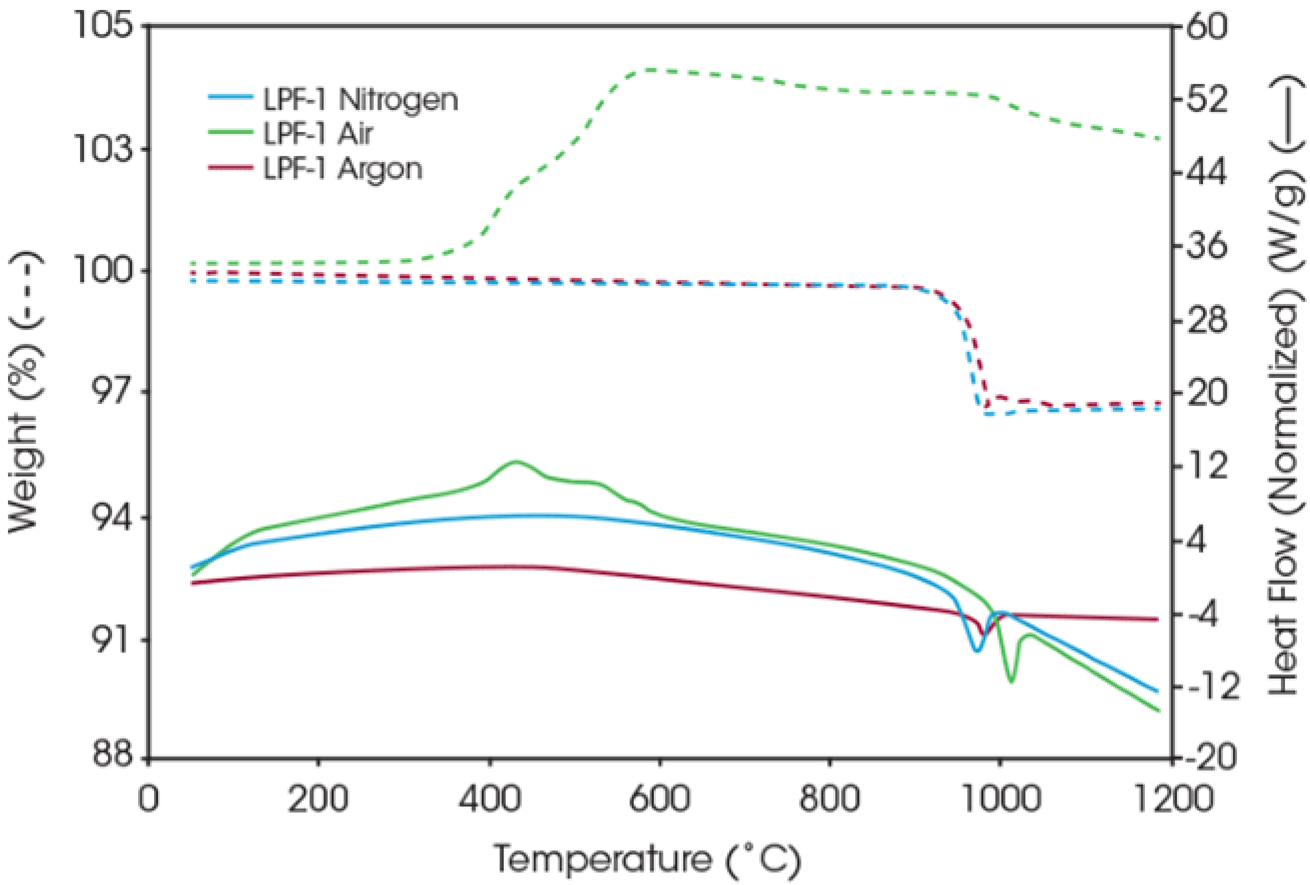

Figure 3. *Coated LFP thermal stability under nitrogen, air, and argon. Image Credit: TA Instruments*

In addition to the nitrogen environment, samples were run in air and argon to assess stability and interactions with those gases. Figure 3 presents the resulting heat flow and weight loss of the coated LFP in air, nitrogen, and argon atmospheres. In nitrogen and argon, the coated LFP remains stable until the sample temperature exceeds 900 °C, at which point the coating begins to degrade.

The coated LFP sample in air exhibits an exothermic reaction peak near 432 °C, along with a weight increase beginning around 300 °C, likely attributable to oxidation. As indicated in Table 1, the peak temperature of the coated LFP is 975 °C in nitrogen and 982 °C in argon.

Table 1. Endotherm peak temperature and weight loss of the coated LFP under nitrogen, air, and argon. Source: TA Instruments

Purge Gas	Peak Temperature of Endotherm (°C)	Weight Loss (%)
Nitrogen	975	3.13
Air	993	–
Argon	982	3.05

SDT enables rapid first pass screening of the temperature stability of LFP. These findings can guide the selection of annealing settings for XRD analysis to assess alterations in LFP crystalline structure after exposure to elevated temperatures. Coated LFP specimens were annealed under nitrogen at 950 °C, where an endothermic phase transition begins in Figure 2.

Additional coated LFP samples were annealed in air at 350 °C due to the observed weight gain around this temperature in Figure 3. This weight increase suggests potential oxidation, and SDT evaluation can be utilized to identify the onset temperature of this reaction. The first derivative weight signal plotted in Figure 4 reveals the rate of weight gain, showing an onset at 325 °C.

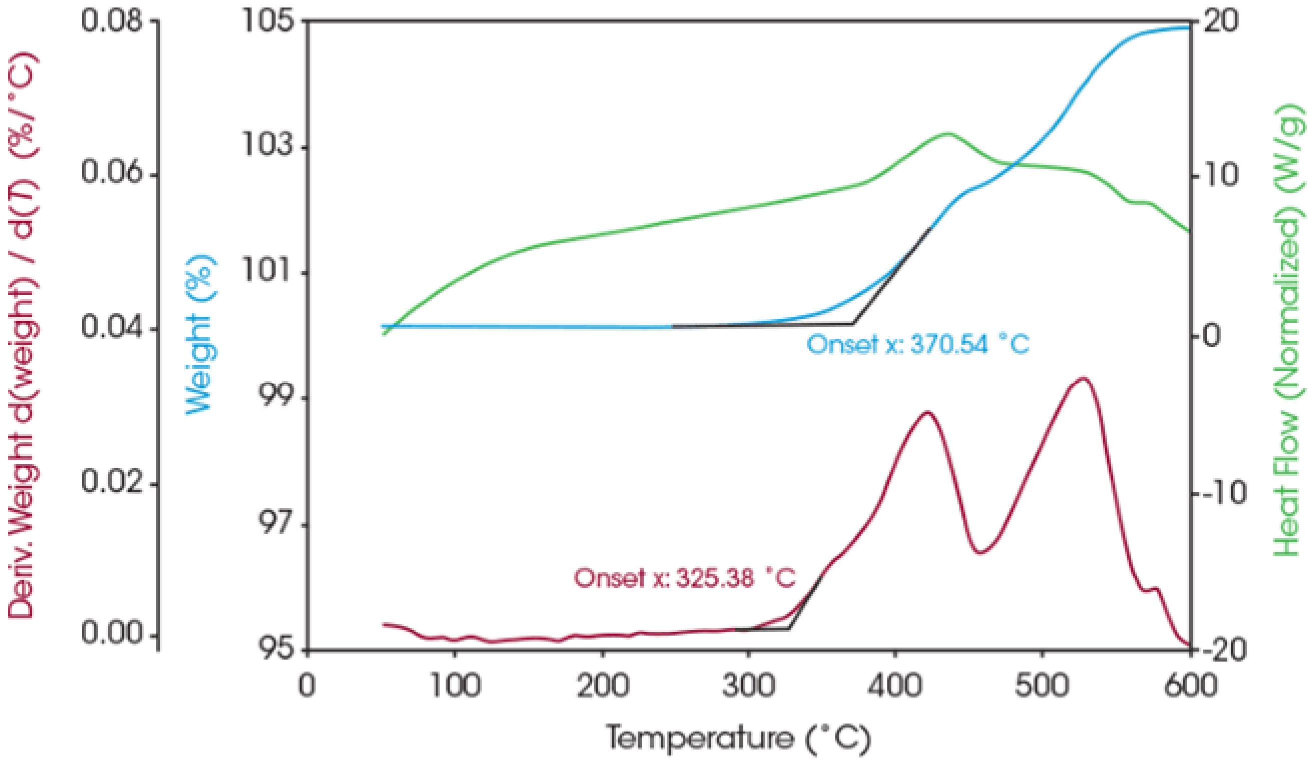

Figure 4. *Weight change (blue), heat flow (green), and derivative of weight change (red) of coated LFP in air. Image Credit: TA Instruments*

Oxidation was verified by the corresponding XRD experiment, illustrated in Figure 5. The impurity phase forms when powders are annealed in air, indicating an oxidation reaction between the coated LFP powders and atmospheric oxygen at 350 °C. No impurity phases were detected in powders annealed under nitrogen.

However, the peaks appear to broaden, which may indicate thermally induced lattice distortion.[5] This broadening could be associated with subtle changes in the crystal structure caused by annealing at 950 °C—the temperature at which a phase transition begins, as suggested by the SDT data shown in Figure 2.

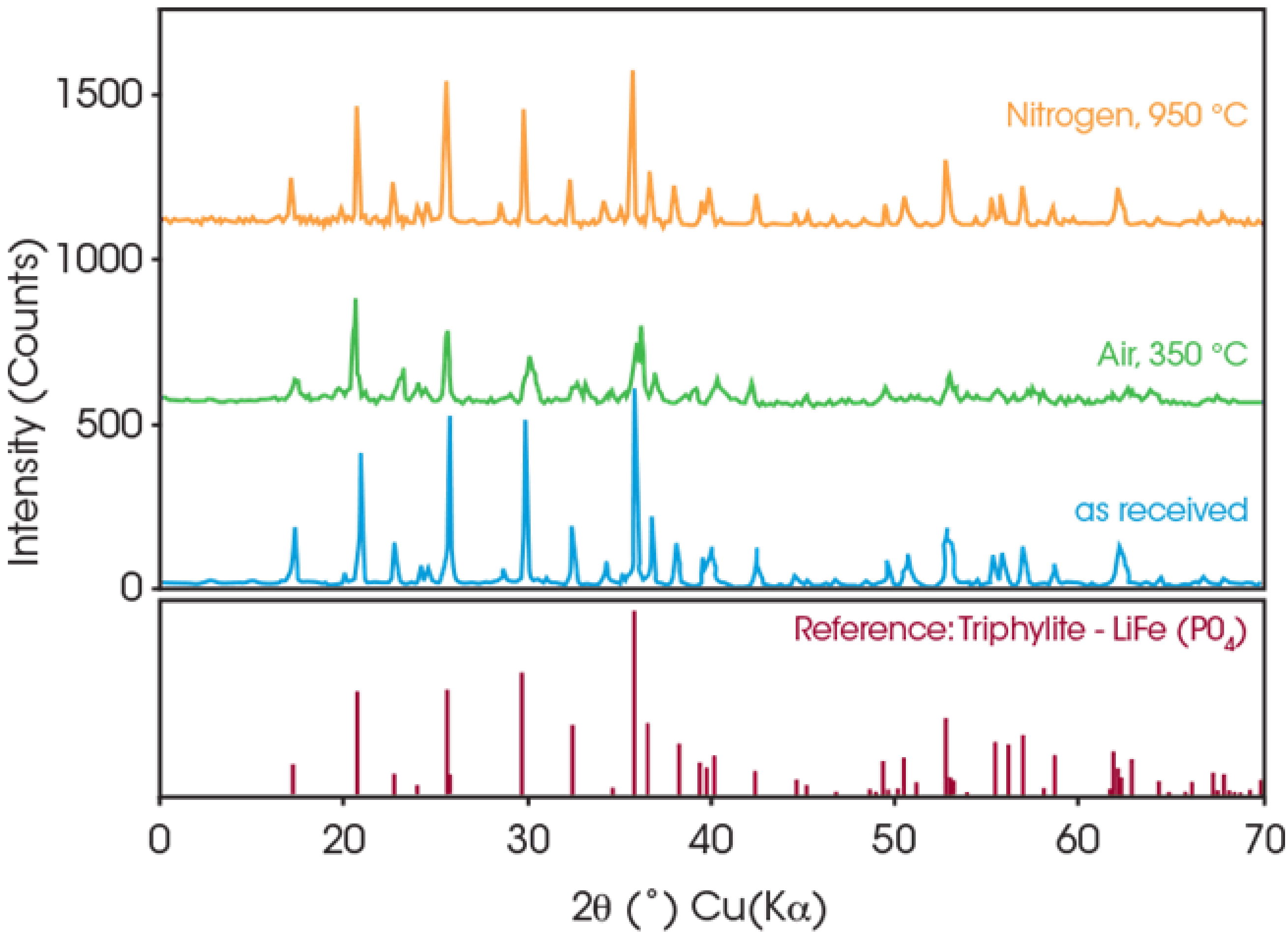

Figure 5. *XRD analysis of coated LFP annealed under nitrogen at 950 °C (orange), air at 350 °C (green), as received (blue), and reference LFP (maroon). Image Credit: TA Instruments*

Conclusions

The TA Instruments SDT is a valuable tool for evaluating the thermal stability and phase transitions of active materials used in lithium-ion battery cathodes. It allows for the investigation of reaction temperatures, energy release, weight changes, and material interactions with various atmospheres, including air, nitrogen, and argon.

In this study, the LFP sample was found to contain approximately 3 wt% of organic coating material. The sample remained stable in nitrogen and argon environments up to 900 °C.

In contrast, samples exposed to air began to oxidize at 370 °C and exhibited a higher phase transition temperature thereafter. This oxidation behavior was further confirmed through XRD analysis.

Beyond providing insights into cathode stability, SDT data can also be used to examine the crystalline structure of LFP at key temperatures, such as those associated with oxidation or phase transitions.

Acknowledgments

Produced from materials originally authored by Jennifer Vail, Ph.D., and Andrew Janisse, Ph.D., Application Specialists at TA Instruments and Hang Lau, Ph.D., New Market Development Scientific Lead at TA Instruments in collaboration with NEI Corporation (Somerset, New Jersey).

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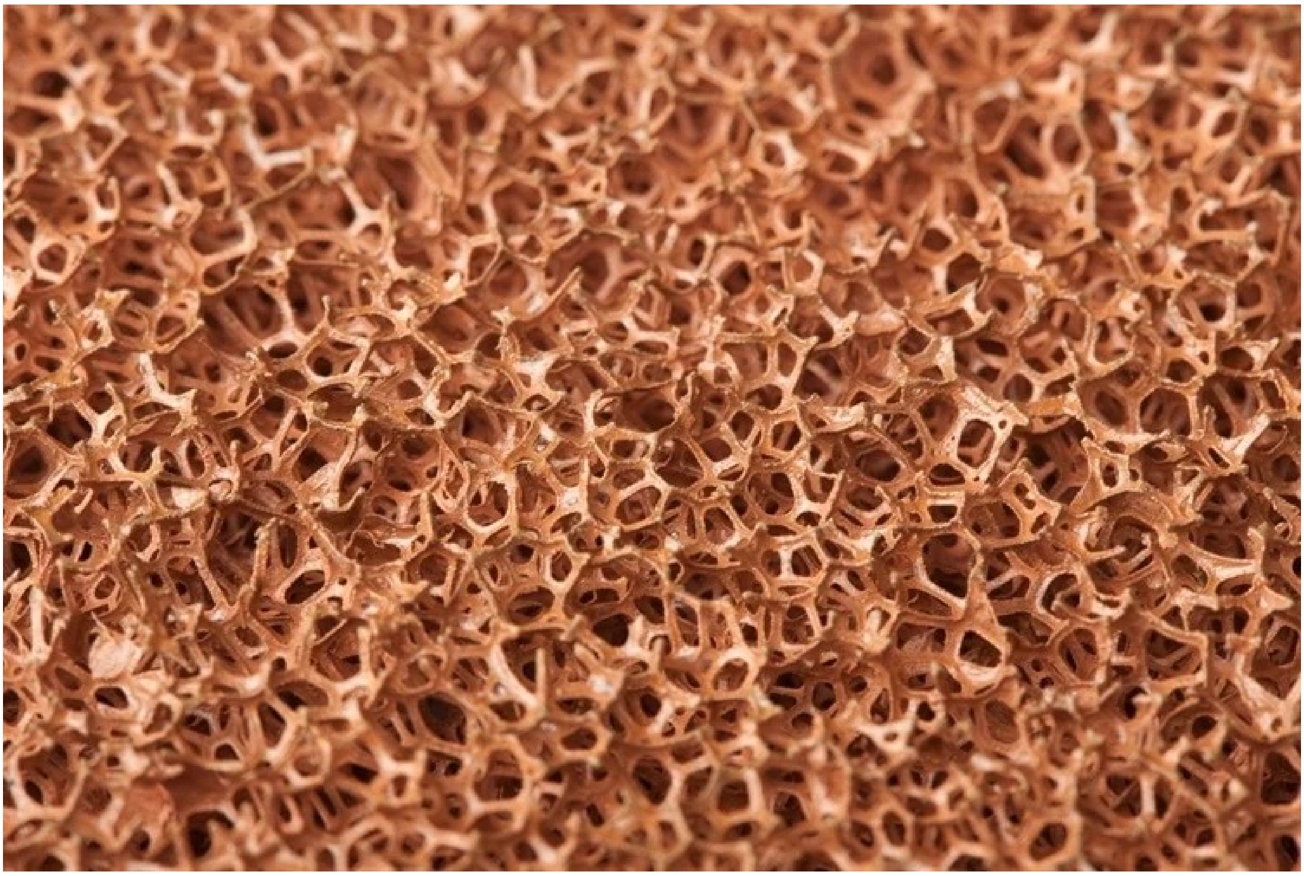
Enhanced Thermal Storage Performance of Copper Foam-Phase Change Material Composites

A recent article in the *Journal of Energy Storage* investigated the impact of non-linear porosity distributions in copper foam on the thermal performance and melting behavior of palmitic acid, a phase change material (PCM). The study used the enthalpy-porosity approach and a local thermal non-equilibrium (LTNE) model to analyze positive and negative porosity gradients in the x and y directions.



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Background

Latent thermal energy storage (LTES) systems using PCMs are effective for energy storage and temperature regulation due to their ability to store and release heat at nearly constant temperatures. PCMs absorb or release energy during phase transitions, but their low thermal conductivity limits heat transfer efficiency.

Incorporating metal foams improves PCM thermal conductivity but reduces storage capacity and increases system costs. Gradient porosity in metal foams offers a potential solution, yet the effects of non-linear porosity distributions on thermal performance remain underexplored. This study numerically analyzed the impact of such porosity gradients on temperature uniformity, melting behavior, and overall thermal storage efficiency of a PCM.

Computational Methods

The study modeled a two-dimensional (2D) 100×100 mm^2 cavity filled with palmitic acid as a PCM and embedded within porous copper foam. Alongside a baseline uniform porosity (UP), the effects of porosity gradients—both positive and negative in the x and y directions—were analyzed. The left wall of the cavity was held at a constant temperature, while the remaining walls were thermally insulated to investigate the influence of porosity distributions on thermal performance.

The phase change characteristics of palmitic acid were analyzed using Differential Scanning Calorimetry (DSC). The phase transition peak, phase transition interval, and latent heat of fusion were derived from the charging curve.

The enthalpy-porosity approach was used to simulate the melting process of the PCM. The LTNE model accounted for heat exchange between the PCM and the copper foam ligaments. Heat transfer between the phases was represented using a convective term to enhance modeling accuracy.

The transient phase change process was simulated using the Finite Element Method (FEM) through COMSOL Multiphysics 6.1 to solve the equations governing the system. The accuracy of these numerical results was examined relative to previously reported experimental results for a 100×100×10 mm^3 enclosure of paraffin-copper foam composite with 97 % porosity.

Results and Discussion

The porosity gradients in different directions had varying effects on the thermal performance of palmitic acid. For instance, a positive porosity gradient along the x-direction considerably decreased melting time relative to the UP case. This was attributed to the enhanced thermal conductivity near the heat source, which reduced conduction resistance.

Notably, lower porosity regions aligned with higher heat flux areas facilitated better heat transfer, enabling faster melting. Among the numerous scaling exponents (n), $n = 0.5$ exhibited the maximum performance enhancement with a 10.34 % decrement in melting time and a 16.39 % increment in the energy storage rate relative to the UP configuration.

A positive porosity gradient along the y-direction enhanced natural convection currents due to higher porosity at the cavity's top. The melting rate increased during the later stages of melting because greater thermal conductivity in the lower zone improved heat transfer and mitigated corner effects. Notably, n = 0.5 demonstrated a 24.49 % improvement in the energy storage rate relative to the UP case.

The optimized 2D porosity distribution with positive gradients in the x and y directions led to a 22.67 % decrement in melting time and a 32.38 % increment in the average energy storage rate relative to the UP case.

Non-linear porosity distributions with positive gradients improved temperature uniformity across the system. Configurations with optimized porosity gradients exhibited more consistent temperature distribution and enhanced thermal performance.

Conclusion

The study analyzed the effects of non-linear porosity distributions on the thermal energy storage performance of PCM-metal foam composites. The investigation focused on a copper foam and palmitic acid system in a 2D domain, evaluating metrics such as energy storage density, melting time, storage rate, and temperature uniformity.

The results showed that a 2D porosity gradient improved heat transfer across different melting stages, leading to a more efficient and uniform melting process. However, the findings are specific to the studied conditions and may differ with variations in parameters such as heat source temperature and location, average porosity, geometrical dimensions, and aspect ratio.

Further research is needed to understand how these factors influence the optimal porosity distribution. Aligning porosity gradients with dominant heat transfer mechanisms during different melting phases could enhance the efficiency of thermal storage systems.

Journal Reference

Kotb, A., Wang, S. (2024). Enhanced thermal storage performance with non-linear porosity distribution in copper foam-PCM composites. *Journal of Energy Storage*. 114612. DOI: 10.1016/j.est.2024.114612, https://www.sciencedirect.com/science/article/pii/S2352152X24041987

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Thermochromic Polymers for Heat Detection Systems

A recent review article in *Polymers* explored recent advancements in thermochromic polymer nanocomposites, focusing on their properties, applications in heat detection systems, and the challenges hindering their broader adoption.

Image Credit: AlexandrBognat/Shutterstock.com

Background

Chromic materials can change color in response to external stimuli, like temperature. Reversible thermochromic polymers, a type of chromic material, are gaining attention for use in heat detection systems.

Understanding the structural, optical, thermal, and mechanical properties of thermochromic materials is crucial for their employment. Despite certain challenges, their adaptability, reliability, and responsiveness are promising for real-time and cost-effective temperature monitoring, with potential applications in energy-efficient building coatings and drug delivery systems.

This article reviews reversible thermochromic nanocomposites for sensor applications.

Reversible Thermochromism

Reversible thermochromic materials change color when exposed to different temperatures and return to their original hue as the temperature normalizes. These color changes occur due to molecular rearrangements, energy level transitions, or particle size variations. Adding chemical components like chromophores can also induce reversible thermochromism.

The specific mechanism behind reversible thermochromism varies by material. For example, crystalline synthesis-induced lattice disruptions alter the absorption spectrum, while changes in molecular mobility cause color shifts in glass synthesis.

Materials like Cu_2HgI_4 and Ag_2HgI_4 change color as their structure shifts from tetrahedral to cubic on heating. Inorganic materials containing Cr^{3+} ions exhibit thermochromism through the ligand geometry mechanism, while coordination number influences thermochromism in inorganic salts containing crystal water. Cholesteric-type reversible liquids change the pitch of their spiral structure with temperature shifts.

Different techniques are used to synthesize reversible thermochromic materials, including polymerization, solution mixing, melt blending, nanoparticle encapsulation, and coating/impregnation. These techniques make thermochromic materials adaptable, responsive, and energy-efficient across different applications.

Understanding these materials is crucial for their diverse applications. Spectroscopic methods like Fourier-transform infrared and ultraviolet-visible spectroscopy can accurately assess molecular vibrations and absorption with temperature. Scanning electron and transmission electron microscopy help observe morphological changes and color variations.

Thermal properties are examined using thermogravimetric analysis and differential scanning calorimetry, while tensile testing assesses robustness and mechanical behavior. Surface analysis through atomic force microscopy and X-Ray photoelectron spectroscopy reveals the materials' long-term stability and color-changing capabilities.

Application in Heat Detection Systems

Utilizing reversible thermochromic materials significantly enhances the operational efficiency of heat-sensing systems. When used in fire alarm systems, these materials ensure safety and mitigate fire-related risks, leading to more robust and adaptive fire safety solutions.

Coatings of liquid crystal thermochromic materials like vanadium dioxide can help develop energy-efficient smart window systems capable of thermoregulation through transparent heaters. Thermochromic materials also ensure safety, quality, and compliance in food processing and packaging. Smart food packaging using these materials allows time-based monitoring, revealing changes over time.

In industries, thermochromic materials enable effective temperature monitoring and process optimization. For instance, reversible thermochromic microcapsules improve color in paper manufacturing.

Thermochromic materials can also monitor temperature and other health parameters in healthcare equipment. Issue-mimicking thermochromic phantoms are crucial for assessing high-intensity focused ultrasound during medical procedures.

Through integration into wearable devices and textiles, thermochromic materials can help detect heat and ensure comfort, signaling dangerous temperatures through color changes.

Advantages and Challenges

Reversible thermochromic materials offer numerous advantages in a wide range of applications. For example, vanadium oxide in smart windows can reduce energy consumption by 9.4 % compared to traditional methods.

These materials are resilient in diverse environmental conditions, seamlessly integrating with various substrates and coatings to enhance overall system efficiency. They are a cost-effective alternative to complex electronic systems, offering clear, easy-to-read displays of temperature changes for immediate recognition. They are highly adaptable, functioning in custom temperature ranges and color changes.

However, maximizing the benefits of thermochromic materials requires addressing challenges such as accurate calibration according to reference temperatures. The sensitivity and response time of some materials also vary with external factors, making it essential to ensure color stability and robustness for practical applications.

Future Prospects

Reversible thermochromic materials exhibit immense potential across various applications. Current research focuses on achieving high-resolution monitoring, creating tailored materials, and integrating these materials into smart systems.

Innovative fabrication methods and eco-friendly materials should be explored for future thermochromic materials. Recyclable materials such as choline hydroxide catalysts help reduce resource use. Precise temperature mapping using micro- and nanofabrication techniques will enable novel applications, such as satellite thermal regulation.

The authors suggest that future research should prioritize expanding color options and reducing reliance on external stimuli to improve the efficacy and applicability of reversible thermochromic polymer nanocomposites.

Journal Reference

Supian, ABM., *et al*. (2024). Thermochromic Polymer Nanocomposites for the Heat Detection System: Recent Progress on Properties, Applications, and Challenges. *Polymers*. doi.org/10.3390/polym1611154

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A Deep Dive into the Thermal Degradation of PET and Recycled PET Fibers

The use of recycled polymer materials is steadily increasing, as manufacturers look for ways to substitute virgin polymers with recycled feedstock. To ensure that this shift doesn't compromise product performance, it's essential to characterize the recycled polymers and compare their properties against their virgin counterparts.

One effective method is thermogravimetric analysis (TGA), which can predict a material's lifespan and help prevent premature failure.

This study investigates the degradation kinetics of both virgin and recycled polyethylene terephthalate (PET) fibers used in 3D printing, using TGA. Results show that the lifetime predictions for the two types of samples are closely aligned.

Introduction

The widespread presence of plastics in the environment has prompted consumers, governments, and companies to seek more sustainable and renewable alternatives.

PET is one of the most commonly used polymers, especially in plastic bottles and packaging across a range of industries.

Due to its extensive use, PET is a major component of plastic waste streams. Fortunately, it's also among the easiest polymers to recycle.[1]

Polymer recycling, however, is a multifaceted process involving primary, secondary, and tertiary techniques.

Primary recycling involves the reuse of uncontaminated, discarded polymer without significant processing. Secondary recycling—also known as mechanical recycling—entails physically reprocessing the material while preserving its chemical structure. Tertiary or chemical recycling uses processes such as pyrolysis or hydrolysis to break down the polymer into its basic chemical components.[2,3]

Of these, mechanical recycling is the most widely adopted, although it comes with concerns, particularly regarding polymer degradation over time and potential declines in material performance.[3]

As a result, it's essential to evaluate material properties and performance when working with recycled PET (rPET) feedstock. Prior research has focused on characterizing rPET in applications such as honeycomb structures, as well as using rheology to assess melt stability and optimize processing conditions.[4,5]

Another key consideration when using rPET is understanding its degradation kinetics in comparison to virgin PET.

TGA offers an efficient alternative to oven aging for studying degradation. TGA tracks weight changes in a material as it's heated at controlled rates, capturing decomposition behavior. The resulting data can be used to estimate a material's projected lifespan and provide insights into its degradation kinetics.6

Experimental

This study examined and compared the degradation kinetics of PET and rPET filaments used in 3D printing.

Commercially sourced samples from a single manufacturer were analyzed. Key degradation kinetic parameters—activation energy and projected lifetime—were determined using TGA following the Flynn/Wall/Ozawa isoconversional method, as outlined in ASTM E1641.[7]

In TGA kinetic evaluation, the conversion (α) of a sample refers to the ratio of actual mass loss to the total mass loss during the degradation process. This value is calculated using Equation 1.

$$\alpha = \frac{W_i - W}{W_i - W_f} \qquad (1)$$

Where:

- W = sample weight at any point during the degradation
- W_i = initial weight
- W_f = final weight

The conversion level may differ from the percent weight loss of a sample run in TGA when residue from nonreactive fillers or char is present. In the Flynn/Wall/Ozawa method, the temperatures in the degradation profile at specific conversion levels for several ramp rates are documented. These are considered the isoconversional temperatures for every conversion level and ramp rate.

After identifying the isoconversional temperatures, plots can be generated to determine activation energy, expected lifetime, and the relative thermal index (RTI). The RTI is the highest service temperature where a critical material property, such as degradation percent, remains above a defined critical threshold over an extended period. This index can be used as a comparison of the thermal endurance of multiple materials.

Measurements were conducted using a TA Instruments™ Discovery™ TGA 5500 with 7 mg (± 1 mg) samples in platinum pans. The samples were heated through decomposition from room temperature to 800 °C under nitrogen purge.

For this study, five different heating rates were utilized: 1 °C/minute, 2 °C/minute, 5 °C/minute, 10 °C/minute, and 20 °C/minute.

Results and Discussions

Figure 1 shows the weight loss profiles of PET and rPET at five different heating rates. Each of these thermal curves can be used to determine the absolute temperature at a constant conversion.

It is worth noting that, due to the inert environment, char residue remains from each sample; however, it is accounted for in the conversion percentage calculation.

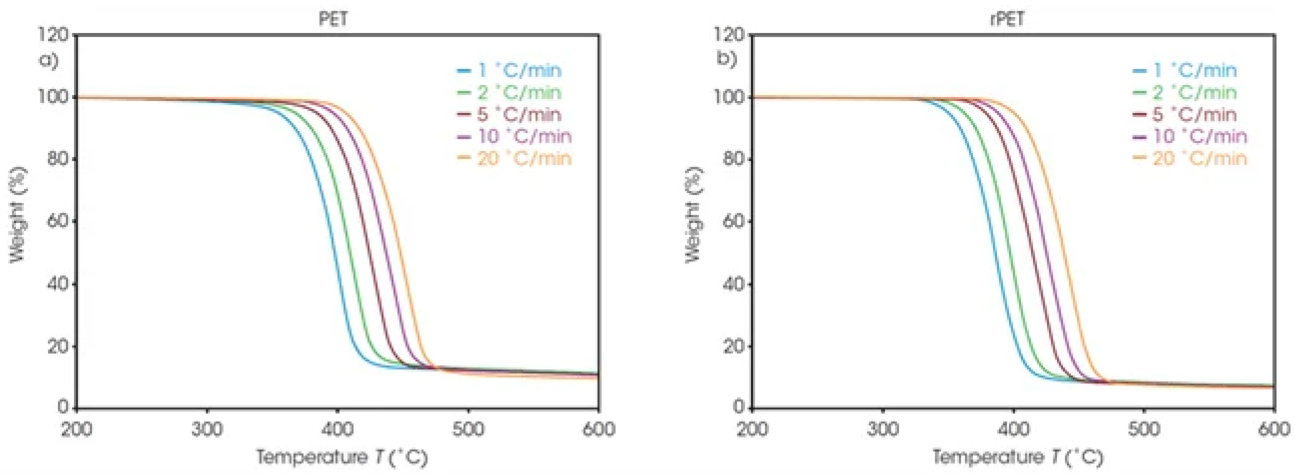

Figure 1. *Weight loss profiles of a) PET, and b) rPET. Image Credit: TA Instruments*

Plots of the logarithm of the heating rate versus the reciprocal of the isoconversional temperatures, shown in Figure 2, are used to calculate activation energy. The slopes of these curves are applied in Equation 2 to iteratively determine the activation energy at each conversion level, following the approach outlined in previous studies.[6]

$$E = \frac{-R}{b}\left[\frac{d\log\beta}{d(\frac{1}{T})}\right] \quad 2$$

Where:

- E = activation energy (J/mol)
- R = gas constant (8.314 J/mol K)
- T = temperature at constant conversion (K)
- b = heating rate (K/min)
- b = constant, approximation derivative (0.457) [ASTM E1641-18]

A "Log Heating Rate" curve can provide valuable insight when analyzing decomposition of a polymer sample. Uniform decomposition mechanisms at all isoconversion levels result in parallel lines with equivalent slopes. At low conversion levels, the slopes may not match due to weight loss from small volatiles escaping the system.

Consequently, analyzing decomposition activation energy is more common at slightly higher conversion levels, where weight loss can be more reliably attributed to sample thermal degradation. A frequently selected conversion level is 5 %.

At a 5 % conversion level, the activation energy for the thermal degradation of PET and rPET is determined to be 210.6 and 213.9 kJ/mol, respectively. This indicates that the polymer degradation is the same for both batches, regardless of whether the material is recycled or virgin.

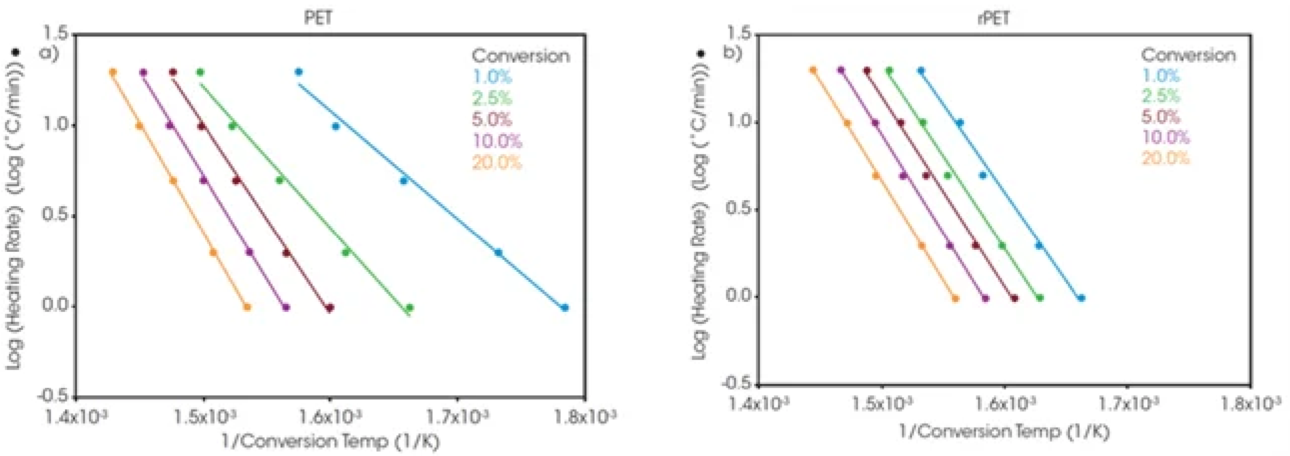

Figure 2. *Log Heating Rate vs Conversion Temperature of a) PET, and b) rPET. Image Credit: TA Instruments*

The calculated activation energy enables determination of lifetime thermal stability as described in ASTM E1877.[8] The resulting plots are illustrated in Figure 3, predicting material thermal stability as a function of time and temperature.

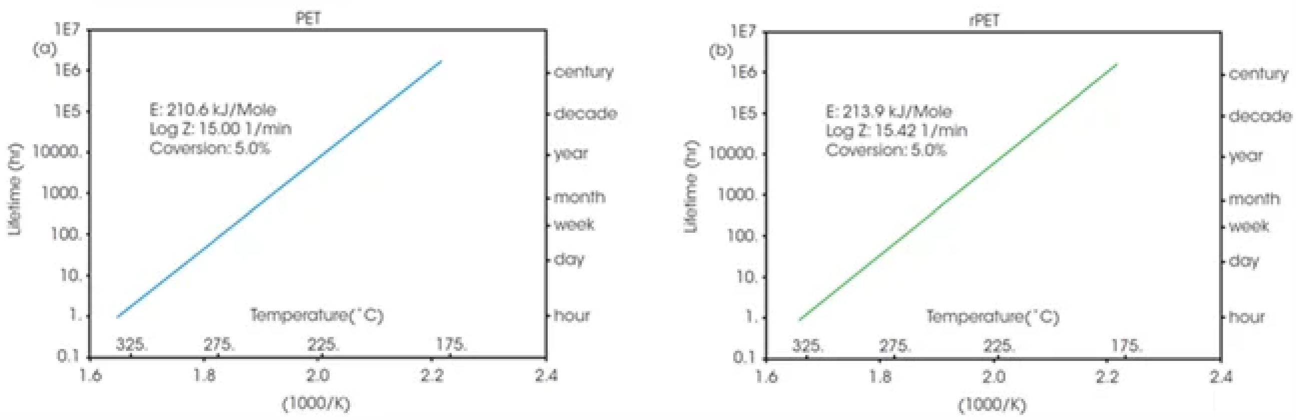

Figure 3. *Estimated lifetime (log scale) vs reciprocal of the failure temperature of a) PET, and b) rPET. Image Credit: TA Instruments*

The RTI for multiple materials can be established by choosing a common thermal lifetime and determining the failure temperature (Tf) for each system to reach the designated conversion over the specified lifetime. Based on Figure 3, the failure temperature for PET to reach 5 % conversion over a five-year period (43800 hours) can be calculated as 208 °C.

The RTI of rPET is functionally the same, calculated as 206 °C over the same lifetime. This shows that products manufactured using material sourced from this recycled feedstock will exhibit the same thermal stability over their lifespan as those manufactured with the virgin material.

Conclusion

A TA Instruments Discovery TGA 5500 was used to predict the lifetime thermal stability of PET and rPET. Several dynamic heating ramps were applied to each sample to identify the kinetic parameters of thermal decomposition by the Flynn/Wall/Ozawa isoconversional method.

The sample composed of recycled feedstock demonstrated thermal stability and lifetime performance comparable to that of the virgin material sample. It is worth noting that, in this case, switching to a more sustainably sourced material does not alter predicted thermal stability.

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Acknowledgments

Produced from materials originally authored by Andrew Janisse, PhD and Jennifer Vail, PhD at TA Instruments.

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Microstructural Changes in Steel Under Various Heat Treatments

Steel is one of the most extensively used alloys composed of iron and carbon and sometimes has other alloying elements for improved and enhanced quality for desired applications.

Image Credit: SasinTipchai/ShuttleStock.com

The properties of steel can be improved in terms of fatigue, weldability, hardness, wear and corrosion resistance, ductility, and tensile strength by either alloy addition or heat treatment processes. This article discusses the effects of heat treatment on steel and the microstructural changes, the equipment and procedures used, and recent relevant studies.

Microstructure of Steel

The microstructure of steel is composed of grains, each with its own crystal lattice structure. Heat treatment subjects steel to controlled heating and cooling processes to alter its microstructure, resulting in changes to hardness, strength, toughness, and other mechanical properties.

The three primary phases of steel microstructure are ferrite, cementite, and austenite, and their transformations play a pivotal role in determining the material's final characteristics.

Heat Treatment Processes

Annealing is a heat treatment process designed to relieve internal stresses, improve machinability, and refine grain structure by heating steel to a critical temperature and then slowly cooling it with control, which allows a finer and more uniform grain structure formation.

In contrast to annealing, quenching is a rapid cooling process that involves immersing hot steel into a cooling medium, like water, oil, or polymer solutions, preventing equilibrium phase formations and resulting in a hardened microstructure characterized by martensite.

Sometimes, after quenching, when the requirement is to have steel with reduced brittleness and enhanced toughness while maintaining a certain hardness, another process called tempering is done by controlled reheating quenched steel to a temperature below its critical point, which decomposes martensite into ferrite and cementite microstructures.

Another heat treatment process similar to annealing is normalizing, which involves air cooling instead of controlled furnace cooling.

In the normalizing process, steel is heated to a critical temperature, held for a specific duration, and then allowed to cool in ambient air, refining the grain structure and enhancing its mechanical properties, making it particularly useful for improving its machinability.

An example of these heat treatment processes is covered in one study that investigated the microstructural changes in AISI 1050 steel under various heat treatments, including annealing, normalizing, and spheroidizing.

Results indicated that heat treatments influenced grain size, yielding altered mechanical properties. Annealing led to increased grain size, reduced strength, and enhanced ductility, while normalization resulted in a slight grain size increase with higher strength. Spheroidizing showed a significant decrease in grain size, yielding softer material with improved ductility.

Equipment to Treat and Analyze Steel Microstructures

The most obvious requirement for the heat treatment of steel is a furnace, usually designed to control the heating process in terms of temperature, humidity, and duration for steel to achieve desired mechanical and metallurgical properties.

Similarly, for analyzing microstructures of steel, several technologies can be implemented, including scanning electron microscopes (SEM), transmission electron microscopes (TEM), X-Ray Diffraction (XRD), and Differential Scanning Calorimeter (DSC).

For instance, a 2019 study used transmission electron microscopy and electron backscatter diffraction (EBSD) for microstructure analysis. The researchers focused on enhancing the microstructure of economical duplex stainless steel with and without tungsten (W) addition to achieve high tensile strength, elongation, and pitting resistance.

The study optimized the heat treatment process to control the comparison and distribution of the two phases (ferrite and austenite).

The highest product of tensile strength and elongation was achieved for Cr19 series duplex stainless steel following solution treatment for five minutes at 1050 °C. The microstructure analysis revealed an excellent transformation-induced plasticity (TRIP) effect, primarily attributed to the existence of a more unstable austenite phase.

Recent Developments

Microstructural Changes in EN353 Steel

In a 2023 study on EN353 grade steel, researchers investigated the impact of heat treatment and microstructural changes on steel properties.

The researchers predicted the continuous cooling transformation behavior of EN353 steel using JMat-Pro software, revealing phases like bainite, perlite, and carbide inclusions in microstructural examinations. Specifically, a specimen of size 40×40×40 mm underwent heat treatment at 870 °C for 2 hours, followed by isothermal heating at 600 °C for 73 minutes and air cooling.

The study addressed the challenges of achieving a fine pearlitic microstructure through normalizing alone, highlighting the necessity of combining isothermal heat treatment for optimal results.

This research has practical implications for industries relying on EN353 steel, as it enhances understanding and control of microstructural changes to improve mechanical properties for various applications, such as gear manufacturing.

Influence of Quenching Temperature on Proto-Austenite in Steel

In another 2023 study, researchers investigated the impact of heat treatment on the microstructure of high-vanadium (V) content quenched and tempered (Q&T) steel. They analyzed proto-austenite grains at different quenching temperatures utilizing metallographic and scanning electron microscopes and studied the precipitation behavior and matrix microstructure characteristics during tempering.

The results showed that higher quenching temperatures increased proto-austenite grain size, with a significant rise beyond 920 ℃.

An increase in quenching temperature also resulted in smaller precipitates, while higher tempering temperatures increased precipitate size. The optimal comprehensive mechanical properties were achieved when quenched at 920 ℃ for 1 hour and tempered at 630 ℃ for 1.5 hours, yielding a tensile strength of 1233 MPa and a low-temperature impact value of 64 J.

This study addresses critical factors influencing steel microstructures, crucial for applications such as mooring chain steel in marine engineering equipment.

Conclusion

The microstructural changes in steel under various heat treatments give rise to changes in the properties of steel.

Different heat treatment techniques, like annealing, normalizing, and tempering, are used for the heat treatment of steel, each having its unique advantage, allowing the steel to have the required properties in accordance with desired applications. These microstructural changes can be observed using analysis tools like SEM, TEM, XRD, and DMC.

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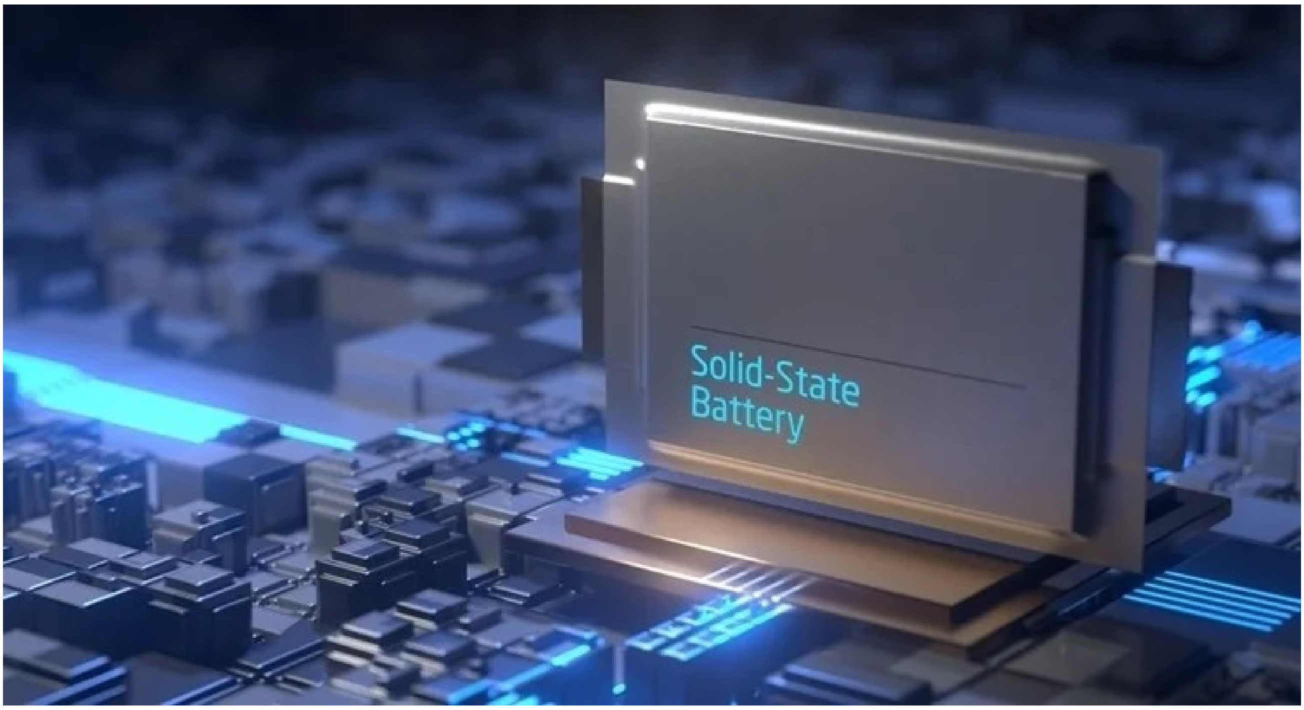
What Are the Latest Innovations in Solid-State Battery Technologies?

Novel battery technology is surpassing current standards. A significant development in this area is the emergence of solid-state batteries (SSBs). These batteries, which use a solid electrolyte, are an improvement over traditional lithium-ion batteries (LIBs) and offer enhanced safety features.



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Researchers in material science are actively working to advance SSB technology. This article explores some of the latest breakthroughs in this innovative field.

Overview of Solid-State Batteries

SSBs use solid electrolytes, unlike LIBs, which use a liquid electrolyte. An advantage of SSBs is enhanced safety, as solid electrolytes remove the risk of thermal runaway and electrolyte leakage.[1] Solid-state batteries also offer higher energy densities, allowing them to store more energy within a smaller footprint.

Portable devices, electric vehicles (EVs), and grid-scale energy storage systems rely on electrochemical power sources like LIBs. However, the commercial LIBs currently in use pose significant safety risks when overcharged since they contain flammable liquid electrolytes.

The energy density of traditional LIBs is also very close to its physiochemical limit. Therefore, the development of technologies with high energy density and intrinsic safety is crucial for large-scale energy-storage systems. As a result, SSBs have seen a resurgence recently for their improved safety and higher energy density.

The transition to SSBs is also promising for addressing challenges in renewable energy storage and EV adoption. By leveraging solid electrolytes, these batteries can withstand extreme temperatures and harsh operating conditions, making them ideal for use in EVs operating in diverse climates.[2]

Key Innovations in Solid-State Battery Technology

Advancements in SSB technology have focused on enhancing the ionic conductivity and stability of solid electrolytes for safer and more efficient energy storage solutions.

Recently, a group of researchers identified high ionic conductivity in pyrochlore-type oxyfluoride, which remained stable in air.[3] This compound exhibited a remarkable bulk ionic conductivity of 7.0 mS cm–1 and a total ionic conductivity of 3.9 mS cm–1 at room temperature (approximately 298 K), surpassing any previously reported oxide solid electrolytes.

The conduction mechanism within this structure involves the sequential movement of Li ions along with changes in bonds with F ions. This discovery not only resulted in the synthesis of a highly conductive and stable solid electrolyte but also introduced a new class of superionic conductors based on pyrochlore-type oxyfluorides.

Considerable efforts have been made to improve polyethylene's low ionic conductivity at ambient temperature. Techniques such as incorporating inorganic fillers to reduce polymer crystallization have been explored.

Since poly(ethylene oxide) (PEO) can coordinate its numerous oxygen atoms with Li-ions, it efficiently facilitates ion conduction within the matrix, making it the most researched polymer in this context. The polymer chains in PEO's amorphous regions are the primary means of ion transport and are crucial for the material's mechanical qualities and conductivity.

The electrochemical properties of PEO have been significantly enhanced by modifying the amount of two distinct liquid crystalline monomers, each with a different length of methylene chain connected to a stiff core and terminal acrylate groups.[4] This modification enhances the structural integrity and ion conductivity of the porous polymer network by forming effective ion transport channels.

Due to their solid-state construction, SSBs have less overall weight and volume, eliminating the need for separators and thermal management systems necessary for liquid electrolyte LIBs (LE-LIBs).[5] This compactness is particularly beneficial for EVs, helping them save weight and space.

Solid electrolytes in SSBs also have a longer lifespan and a slower rate of capacity reduction over time because they are more stable and degrade less under cycling circumstances. Studies in this area have produced materials whose ionic conductivities are either as high as or higher than those of their liquid equivalents.[6]

Compared to liquid electrolytes, which tend to degrade over time and under heat stress, solid electrolytes found in supercapacitors are less susceptible to degradation. Researchers have found that the inherent stability of solid electrolytes helps SSBs last longer, which lowers the need for frequent battery replacements and, over time, lessens the environmental and economic impacts of battery disposal.[7]

Since SSBs have no liquid components, more design flexibility is available. This enables the production of batteries in sizes and configurations that were previously impossible, creating new opportunities for integrating batteries into various products and applications, from wearable electronics to renewable energy sources.[8]

Challenges to Commercialization

Despite the many benefits SSBs provide, several challenges must be addressed before they can be produced on a large scale.

Firstly, the production of SSBs involves complex manufacturing processes that are currently difficult to scale. It requires precise engineering and management to fabricate tiny, flawless layers of solid electrolyte and ensure ideal contact with the electrodes. A major challenge for making SSBs commercially viable is scaling these techniques to mass production while maintaining quality and consistency.

Additionally, the thermal management of SSBs remains a challenge despite their inherent safety and stability at high temperatures, particularly in high-power applications such as electric vehicles. Compared to liquid electrolytes, solid electrolytes have a less effective heat-dissipation capacity. For SSBs to function correctly and have a long lifespan, heat management during fast charge and discharge cycles must be carefully considered in the design.

Lastly, many solid electrolytes, especially those made of ceramic, are brittle, making them difficult to handle and more prone to failure. It is imperative to develop solid electrolytes with sufficient mechanical strength to endure these shocks.

Future Outlook for Solid-State Batteries

As research endeavors persist in pushing the boundaries of ingenuity, addressing pivotal challenges such as manufacturing scalability, thermal regulation, and mechanical resilience, SSBs are set to significantly impact the transition toward cleaner and more sustainable energy systems.

With continual strides in materials science, battery architecture, and production methodologies, SSBs are anticipated to increasingly rival conventional LIBs, offering enhanced safety profiles, augmented energy densities, and protracted operational lifespans.

As collaborative efforts within the sector increase, the widespread commercialization of SSBs holds the potential to drive significant advances toward a more eco-friendly and efficacious energy landscape.

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