TMG News

September 2021

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Material Characterization Tier III – (Part 3 of 3)

Richie Anfinsen

In the second part of this series, we explored the second tier of material characterization. At this level, a designer will have a much better chance of selecting a material based on data sheet values for their application. The second tier of material characterization is even more important now with the higher level of polymer supply issues making alternative material selection an all-to-familiar issue in the industry. However, this level of characterization will miss several important pieces of data on the additives utilized to modify the properties of the material for exposure conditions. At the second tier, we will not understand if an unknown material has antioxidant additives for stability (e.g., hindered phenols for processing), flame-retardant additives, or UV stabilizers to allow for better long-term performance of the parts. This is where we need additional tests from a Tier III characterization to help us understand the additives that will be of great importance to the performance of the part in the expected environment.

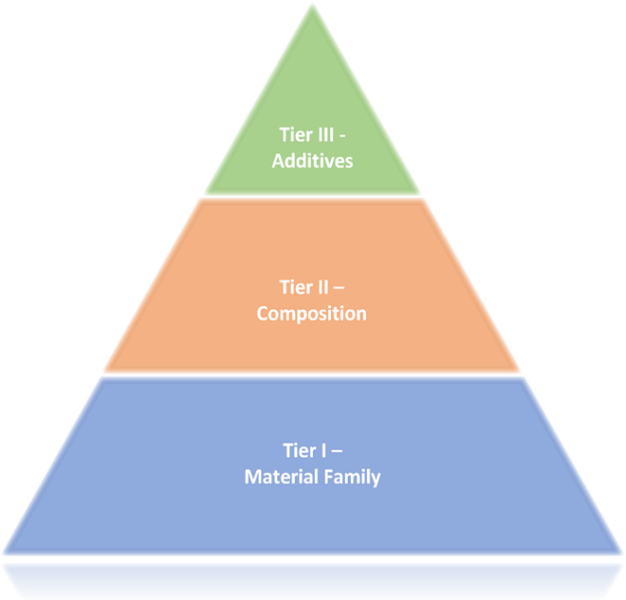

Figure 1. Diagram showing the levels of characterization and the frequency that the analysis level is utilized in a material characterization.

Tier III of material characterization is typically reserved for detailed characterizations of the lower concentration additives that shape the special properties of a material. These additives are typically under 1% by weight within the material and are incorporated into the resin to ensure proper performance over the lifetime of the part. These additives would include antioxidants (to help with processing stability and environmental exposure), slip agents for molding, tackifiers for rubber materials, compatibilizers for blends, flame-retardant additives, and many others. Typically, this level of characterization is employed by designers to reverse engineer materials, resin suppliers, or in cases where part failure has occurred prematurely due to some environmental conditions.

The primary techniques utilized in a Tier III characterization could include mass spectroscopic techniques (GC/MS, LC/MS, HPLC), trace elemental analyses (XRF and ICP), nuclear magnetic resonance spectroscopy (NMR), and gel permeation chromatography (GPC). This is certainly not a comprehensive list of all the higher-level techniques that are utilized to understand the additives in the material. Other techniques can be utilized to meet regulatory standards (RoHS, USP VI, etc.). These tests should be discussed on a project-to-project basis. However, these are the "workhorses" of identifying additives that are commonly used in polymer applications.

The mass spectroscopy techniques are utilized to look at antioxidants, flow aids, tackifiers, and other lower molecular weight substances. This technique provides a spectrum that is used for identification of chemical compounds at

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very low (PPM) concentration. Shown in **Table 1**, are typical results you can expect from this type of analysis. The analysis provides a list of chemical compounds that were in the detectible range of molecular weights for each technique. If you are just running the sample, this is typically just a qualitative identification with limited ability to quantify the amounts of the additives. Quantification is possible with a calibration experiment to a known reference of the additives identified in the material. However, practical designers will rely on material suppliers to determine the correct amount of lower-level additives in a material and testing on the back-end to determine if enough additives are present for the expected exposure and lifetime needs.

Observed RT (min)	Component name	Observed m/z	Expected mass (Da)	Observed mass (Da)	Mass error (mDa)	Response
2.42	1,324- Bis (3,4-dimethylobenzylideno) sorbitol (DMDBS)	415.2113 $(M+H)^+$	414.2042	414.2040	-0.2	90,820
4.62	Ultranox 626 Diphosphate	637.3051 $(M+H)^+$	636.2981	636.2978	-0.3	1,656
5.52	Unidentified	934.6405	--	916.6064	--	67,703
5.62	Irganox® 3114 FF	801.5529 $(M+NH_4)^+$	783.5186	783.5190	0.4	381,998
5.78	Unidentified	1138.7538	--	1120.7200	--	23,167
5.97	Alkanox P-24	605.3146 $(M+H)^+$	604.3083	604.3074	-0.9	6,944
6.07	Irganox 1010 Oxidation Product	1192.8011 $(M+NH_4)^+$	1174.7684	1174.7672	-1.2	86,708
6.13	Irganox 1010	1194.8174 $(M+NH_4)^+$	1176.7841	1176.7836	-0.5	1,395,278
6.14	Cyanox® LTDP	515.4131 $(M+H)^+$	514.4056	514.4058	0.2	158,680
6.96	Irgafos 168 oxidized	663.4536 $(M+H)^+$	662.4464	662.4464	0.0	1,197,690
7.86	Irgafos 168	647.4593 $(M+H)^+$	646.4515	646.4520	0.5	670,076

Table 1. Table of results from an LC/MS test of a thermoplastic elastomer. The results show that the material had a stabilizer package with multiple different antioxidants.

Two of the most popular techniques for identifying and quantifying the elemental make-up of a material are ICP and XRF. In the context of a Tier III characterization, these techniques are typically utilized to help with identification of some flame retardants and stabilizers. For example, the use of ICP and XRF can help to identify if a copper iodide stabilizer is being utilized in a polyamide material.

Nuclear magnetic resonance spectroscopy (NMR) is a technique that is basically an FTIR test on steroids. While FTIR is useful to tell what type of bonds are in a material, NMR can construct the backbone structure and determine where in the polymer backbone, those bonds are located. This makes NMR a powerful technique to deconstruct complex molecules such as thermoplastic urethane elastomers and other thermoplastic elastomeric materials. Additionally, it can provide information on polymer blends and branching, which can be extremely important to polymer performance. It should be noted that a deeper level of knowledge of molecular structure is required to properly interpret these results.

GPC testing is utilized to provide an exact measurement of the molecular weight of a material, including information on the distribution of polymer chain lengths, **Figure 2.** In a Tier III analysis, GPC analysis would be used to identify more performance -based metrics of molecular weight. Specifically, this method is useful to compare the molecular weight distribution, which will

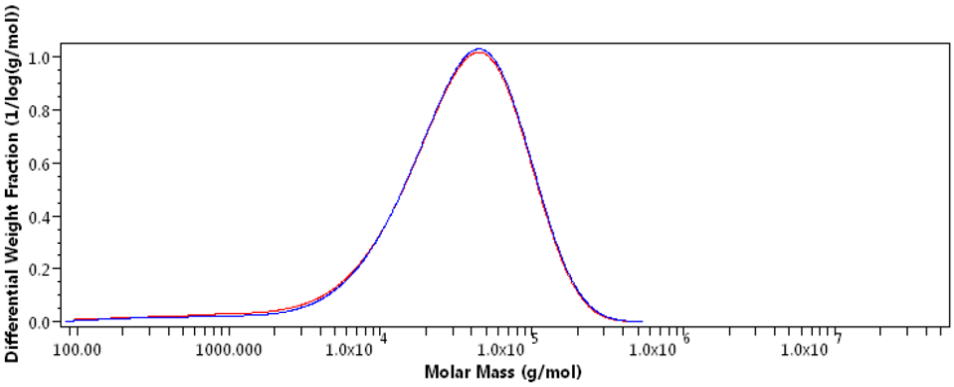

Figure 2– Molecular weight curves for two materials that showed minimal differences in molecular weight and molecular weight distribution. In a material characterization setting, these results would show that these materials are similar in molecular weight and would be expected to have similar properties if all else is equal in the material composition.

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affect the processability and mechanical performance of a polymeric material. In general, this technique is best applied as a comparative analysis between two materials as there is little published data on exact molecular weights of commercial polymers. However, if you need to investigate the molecular weight curve due to concerns about performance, GPC can be used over MFR to investigate the material.

While these are certainly not all of the techniques that can be used to identify polymeric materials, these techniques are definitely the most utilized techniques in material characterization. The next time you have a question of what material is or should be used within a product, take a minute to think about how involved your analysis needs to be. Whether you need a lower-level identification or a complete material deformulation, there are techniques that can help you move your project forward. The Madison Group has significant experience with all levels of material characterization and would be happy to assist you with all of your testing needs.

Click here to read Part 1.

Click here to read Part 2.

Information regarding additional case studies can also be found at:
https://www.madisongroup.com/case-studies.html

If you are interested to have The Madison Group provide training to your team, please feel free to contact us at info@madisongroup.com.

Training Topics

- Failure Analysis
- Plastic Material Selection
- Plastic Part Design
- Moldflow
- Other

Announcements

TMG – Industry News

The Madison Group is excited to offer our training for all Autodesk Moldflow products, both Insight and Advisor.

The need for optimizing our plastic part designs, processes and mold designs prior to first shots, is more critical than ever. Autodesk Moldflow has multiple products to help assist and optimize your project at any stage. Whether you are a part designer that is interested in better understanding your externally provided Moldflow reports, a user that is looking to take full advantage of the tools you already have, or explore what additional tools are available to take you to the next level, we have a training package that can help you accomplish just that.

The Madison Group has a training plan option for any circumstance and budget.

Choose any of the following options:

- **On-site Training**
- **Remote Instructor-Led Training**
- **Private Training**

Benefits of Investing in Moldflow Training

- Keep up to date on the newest solvers and simulation tools for all the Autodesk Moldflow Products designed to save you time.
- Improve your results interpretation skills and help optimize your design.
- Increase your internal knowledge quickly and economically to improve communication and create a culture of innovation.
- Explore additional simulation capabilities to improve overall customer satisfaction.

Benefits of Remote Instructor-Led Training Sessions:

- Allow any of your employees to gain the training without being out of the office.
- Eliminate travel costs so you can have more employees trained.
- Choose interactive, live, instructor-led classes for one-on-one assistance with solver set-up and results interpretation.
- Installation of software not needed prior to training opportunities.

Find a listing of all of our Upcoming Training Sessions here.

Upcoming Educational Webinars #TMGPLASTICEDU

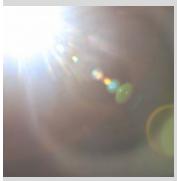
Tuesday, October 12, 2021 - Jack DeSousa
"Plastics In" Series: Appliance Applications
10:00 AM (CST)

The use of plastic materials in electronic appliances has been a growing market. This webinar will introduce the types of plastics being used in these applications, review common design considerations when choosing a plastic material, and go over case studies showing what can go wrong when plastics are not properly implemented.
Click here for more information.

Tuesday, October 26, 2021 - Melissa Kurtz
Plastics Sustainability – Introduction
10:00 AM (CST)

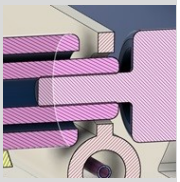
Plastics have enabled innovations in numerous industries. With their popularity come concerns surrounding end of life. As the desire for sustainable products grow, so does confusion on what sustainability means. This webinar will define the language surrounding plastic sustainability and take an in-depth look at a variety of approaches. Click here for more information.

Tuesday, November 9, 2021 - Javier Cruz
The Degradation of Plastics
10:00 AM (CST)



Nothing lasts forever and degradation is just one of the many mechanisms that dictates the end of life for plastic products. This webinar will introduce the concept of degradation and present different mechanisms that can lead to degradation. In addition, techniques for identifying and preventing degradation will be discussed.
Click here for more information.

Tuesday, November 30, 2021—Erik Foltz
An Overview of DFMEA Considerations for Common Secondary Processes for Plastic Assemblies
10:00 AM (CST)



Many presentations have been given that highlight the fundamentals of proper plastic part design. However, an individual plastic part is rarely used as a stand-alone product. This presentation will highlight how selection of common secondary joining operations could influence your part design, and material selection considerations.
Click here for more information.

Tuesday, December 14, 2021 - Jeffrey A. Jansen
The Structure and Properties of Polypropylene
10:00 AM (CST)

Polypropylene is a versatile thermoplastic that can be processed through a variety of techniques and utilized in a wide range of applications. This webinar will provide attendees with an understanding of the properties of polypropylene as well as their dependency on molecular weight, crystallinity, comonomer content and tacticity.
Click here for more information.

Information regarding upcoming educational opportunities can also be found at:
http://www.madisongroup.com/events.html

Webinars/Seminars

Webinar Series

Failure of Plastics
Session 1: Wednesday, October 13, 2021 - *Jeffrey A. Jansen* – SPE
Click here for more information.
and
Session 2: Thursday, October 14, 2021 – *Jeffrey A. Jansen*– SPE
Click here for more information.
Noon-1:00 pm (CST) (both days)

This 2-part webinar series will cover a considerable range of topics important in understanding, diagnosing, and preventing plastic component failure. The most efficient and effective approach to plastic component failure is by performing a systematic failure analysis. Someone once said, "if you don't know how something broke, you can't fix it," and this certainly highlights the importance of a thorough understanding of how and why a product has failed. This webinar series will introduce the attendees to information they need to gain this understanding.

The material covered will include:

- Essential knowledge of why plastic components fail.
- The five factors affecting plastic part performance.
- The process of conducting a failure investigation and methods for understanding how and why a product has failed.
- The importance of ductile-to-brittle transitions and their role in plastic component failure.
- The major plastic failure mechanisms.
- Failure analysis case studies.

The webinar series will focus on practical problem-solving techniques and will utilize case studies to illustrate key aspects of plastic failure and prevention. Participants will gain a better understanding of why plastic components fail, and how to avoid future failures by applying the knowledge learned.

Thursday, November 11, 2021 – *Jeffrey A. Jansen*– SPE
Plastic Datasheets: What They Do and Don't Tell Us
Noon-1:00 pm (CST)

UL Prospector lists tens of thousands of different plastic resins. When tasked with material selection, 99.44% of us turn to the typical property data sheet. Those of us who have been around a while are careful not to call it a "technical data sheet." What are the issues with the single point numbers listed? Why does that often lead to a failed product? What should we be doing instead?

Click here for more information.

Information regarding upcoming educational opportunities can also be found at:
http://www.madisongroup.com/events.html

The Madison Group Teaches Failure Analysis, Design & Prevention

Monday-Wednesday, October 25-27th, 2021
Via Zoom—Live—Online
Presenters: *Jeffrey A. Jansen, Dr. Antoine Rios, Dr. Javier Cruz, and Erik Foltz*
Plastic Part Failure: Analysis, Design & Prevention
8:00 am—4:00 pm (CST)

The University of Wisconsin—Milwaukee School of Continuing Education is offering an online 3-day course entitled, **"Plastic Part Failure: Analysis, Design & Prevention"** taught by The Madison Group Engineers. The course will cover a broad range of topics essential to understanding and preventing plastic failure.

Dive into a broad range of topics essential to understanding and preventing plastic failure. The most efficient and effective approach to plastic component failure is performing a systematic failure analysis following scientific method. Someone once said, "If you don't know how something broke, you can't fix it," highlighting the importance of a thorough understanding of how and why a product has failed.

Benefits and Learning Outcomes:
Get introduced to the strategies behind analysis, design and prevention with course material that includes:

- Learn the essentials of why plastic components fail.
- Understand the five factors affecting plastic part performance.
  - Material, design, processing, installation, and service
- Learn the process of conducting a failure investigation.
- Know the importance of ductile-to-brittle transitions and their role in plastic component failure.
- Understand how and why a product has failed.
- Explore approaches to more quickly respond to and resolve plastic component failure.
- Learn methods and techniques to avoid future failures.

Outline:

- **Introduction to Plastics**
  - -Overview of Plastic
  - -Composition
  - -Properties
  - -Plastic Part Failure
- **Failure Correction and Prevention**
  - -Part Design
  - -Mold Design
  - -Material Selection
  - -Processing
  - -Validation Testing
- **Failure of Plastics Overview**
- **Failure Mechanisms**
- **The Roles of Multiple Factor Concurrency and Statistical Distribution in Plastic Part Failure**
- **Failure Analysis**
  - -Problem Solving / Investigation Techniques – FA and RCA
  - -Failure Analysis Test Methods
  - -Case Studies

CEUs: 2.0, **PDHs:** 20
Enrollment Limit: 30
Program Number: 4830-13805
Registration Deadline: Oct 22

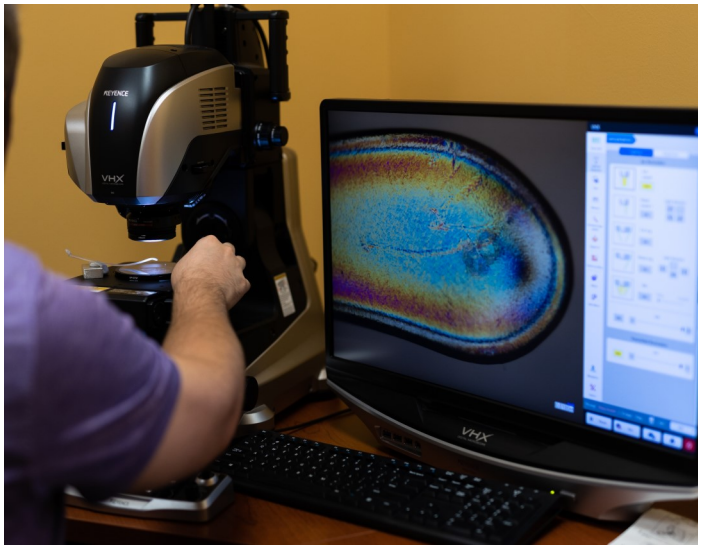
Click here for more information.

Information regarding upcoming educational opportunities can also be found at:
http://www.madisongroup.com/events.html

Through the Lens of The Madison Group

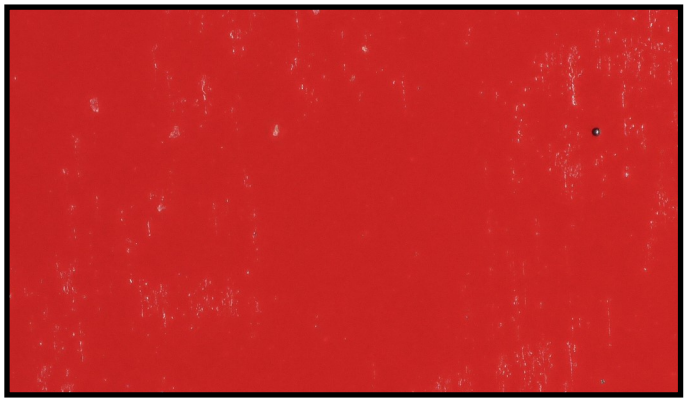
The Madison Group's acquisition of a new state-of-the-art Keyence High-Definition Digital Microscope VHX-7000 greatly expands our analysis capabilities with:

- Automatic 3D imaging to permit the examination of rough and deep fractures.
- High-magnification capabilities (up to 2500x) to permit measurements of coatings and films.
- Advanced illumination options of backlighting, multi-angled oblique lighting, polarization and an optical shadow effect mode that allows the visualization of crack features and textures not visible in a SEM.
- Stitching of high-magnification images to provide a large field of view to improve visualization.
- Remote/virtual capabilities to provide our customers with access to inspections and analysis.

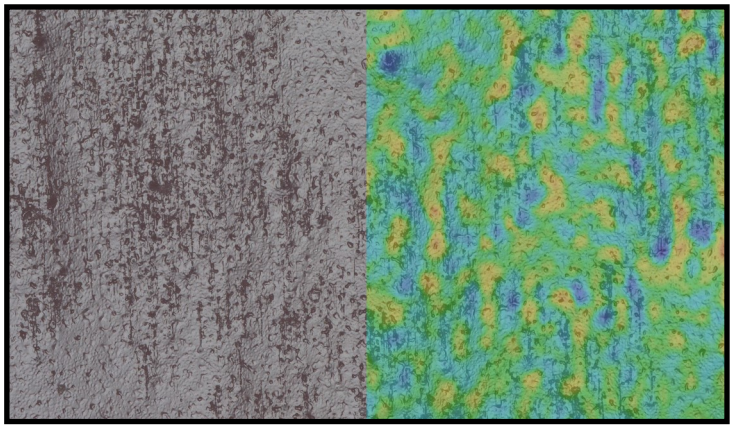


New Keyence Microscope

With the advanced capabilities of The Madison Group's Keyence High-Definition Digital Microscope, the surface metrology of molded polymeric materials can be evaluated. For instance, the dynamic 3D analysis tools of the system permit the surface features to be evaluated on molded parts. Consider the red polyurethane surface shown to the left at 200x. At that magnification, the surface appears glossy and without any discernable features. However, when imaged using the enhanced 3D stacking with full coaxial lighting, the finely dispersed surface particles become visible as shown below. Furthermore, the analysis includes mapping tools that can be extended to provided color contours of the surface heights for the texture. Such analyses can then be used to help diagnose friction and wear issues or to evaluate processing-induced differences in the surface metrology.



Enhanced 3D View (200x)



Polyurethane Surface (200x)

If you are interested to learn more about the capabilities that we have at The Madison Group, please feel free to contact us at info@madisongroup.com.

