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Material Science and more…

Director's note

Dear Reader,

I am proud to continue publishing the Shimadzu Journal. This journal is a compilation of various collaborative research projects and technical reports and applications from our library. In accordance with our corporate philosophy "Contributing to Society through Science and Technology," we conduct our business to meet customers' needs in a wide variety of fields all over the world, providing the cutting edge of technologies. Our brand statement, "Excellence in Science," reflects our desire and sincere and earnest attitude to listen to our customers' requirements and to offer valuable solutions.

The Analytical and Measuring Instruments Division offers state-of-the-art solutions in a wide variety of fields that impact the health and lives of people. Our diverse product lineup ranks as the best in the industry. This lineup includes mass spectrometers, chromatographs, life science instruments, spectrophotometers, surface analytical instruments, microscopes, environmental monitors of exhaust gas and water, strength and fatigue testing machines, X-ray inspection devices, balances, and thermal analyzers.

As a manufacturer, we believe that collaborating with researchers is the best way to develop new solutions that will deliver true contributions to the world. Therefore, in this Journal, we introduce collaborative research projects and share the latest information and results.

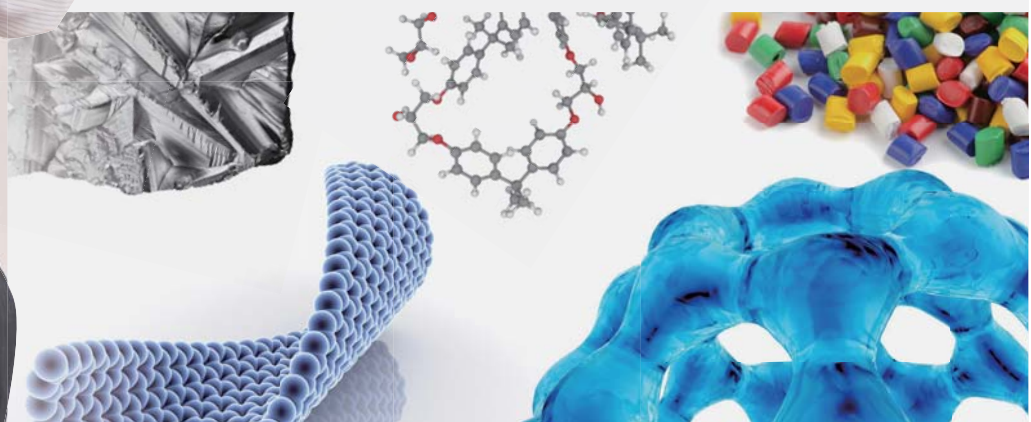
This issue focuses on Material Science and contains results from two collaborations. One is with Professor Frank Walther of TU Dortmund University in Germany and the other with the Japan Aerospace Exploration Agency (JAXA). Both use our testing machines and analytical instruments. In addition, this issue contains information on other applicable topics, as well as the latest news and applications.

We want to establish a good partnership with you. As stated in our corporate slogan of this year, "Best for our customers: Challenge without limits," we are striving to exceed your expectations with the highest technological capabilities and most meaningful solutions. I hope that this journal will be of great help to all of you.

Yours Sincerely,

Teruhisa UEDA, PhD.

General Manager, Analytical & Measurement Instruments Division



CONTENT

Featuring Material Science

Prof. Frank Walther of TU Dortmund University

We interviewed Professor Dr. Frank Walther, Head of Department of Materials Test Engineering (WPT) at TU Dortmund University in Germany. His research is focused on microstructure-based materials and components testing, destructive and non-destructive testing and measurement techniques as applied to characterize structure-property relationships, evaluating the fatigue performance, failure mechanisms and damage processes and predicting remaining fatigue life. He and Shimadzu started collaborative research with first contact in October, 2011. A number of discoveries and achievements have been made with Shimadzu testing machines and analytical instruments such as Autograph (AGS-X, AG-X) and Servopulser (EHF) systems, micro magnetic (MMT) and ultrasonic fatigue testing (USF) systems, ultra-micro hardness tester (DUH), and so on.

Shimadzu:

Professor Walther, thank you very much for spending some time for this interview. Firstly, could you tell us the background of this collaborative research? Why did you choose Shimadzu as your partner?

Prof. Walther:

My research group investigates basic- and application-oriented topics in the field of destructive and non-destructive materials and components testing. Microstructural and mechanism-based characterization of the Low-Cycle-Fatigue- (LCF-) to Very-High-Cycle-Fatigue- (VHCF-) behavior is the key subject. Main modules of the research strategy are the analysis of materials and microstructures, the application of process- and product-optimized measurement techniques for characterization as well as modeling of properties and calculation of lifetime. The exact assessment of the influence of production and in-service conditions on basic material parameters leads to optimized property profiles and enhanced functional safety of components. The portfolio of research ranges from scientific fundamental research to applied (bilateral) development and analysis services for industrial companies.

When I established the Department of Materials Test Engineering (WPT) in December, 2010 at TU Dortmund University, I had nearly no laboratory equipment. Within the context of lab expansion and network building, I contacted the Product Manager for Material Testing Systems at Shimadzu Duisburg, Germany. This was the first contact with Shimadzu, which I did not know before. On the basis of a good understanding and successful implementation of initial projects, a successful collaboration was initiated.

During the installation of initiatory Shimadzu materials testing systems in WPT lab, the quality of the devices and the well-trained service engineers attracted my attention. In addition to these profound impressions, the distance between Duisburg and Dortmund is only 50 km, so that mutual visits in Shimadzu or WPT lab are possible with very less effort.

Shimadzu:

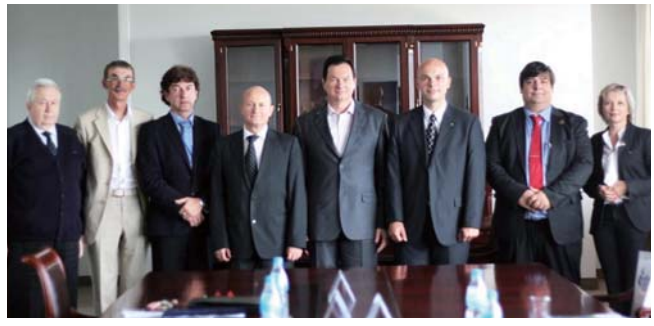
Then could you outline the research and let us know which discoveries and achievements have been made so far?

Prof. Walther:

First of all, development of a well-organized WPT laboratory infrastructure was realized. On the basis of my 15+ years' experience in Materials Testing, I started to evaluate Shimadzu devices and was convinced of hardware and software quality and capability. All these initiatives were appreciated and supported by Shimadzu Europa GmbH.

Picture 1: Prof. Walther at Shimadzu Headquarter Kyoto, Japan

In the context of increasing collaboration for the exchange of experiences, a meeting was organized at Shimadzu's headquarter in Kyoto, Japan (Picture 1). There, substantial fruitful discussions took place and many promising further hardware and software developments were comprehended. Within the initiative to build up a Shimadzu network with reference customers, an additional visit at FEFU University in Vladivostok, Russia, took place (Picture 2). With the support of the Rector of FEFU University, it was decided by the colleagues Prof. Gridasov and Prof. Pogodaev, to work closely together e.g. in DFG (German research foundation) research projects.



Picture 2: Prof. Pogodaev (left), Prof. Gridasov (second from the left), Prof. Walther (third from the right) at FEFU University, Vladivostok, Russia

During my visit to Russia, there was also a Shimadzu fair taking place where I presented my results obtained in industrial research projects carried out using Shimadzu systems (Picture 3).

From the current year, I'm acting as a Visiting Professor at FEFU University. Besides the presentation of a fatigue course, an exchange of PhD students was agreed upon. The first PhD candidate from FEFU visited WPT in December, 2013 for experimental investigations concerning the fatigue performance of glass-metal composites.

In the meantime I visited many colleagues working together with Shimadzu in Germany and Europe. In January, 2014, I was invited for a Shimadzu International Sales Meeting to illustrate the basics of fatigue and lifetime prediction to sales representatives.

As I know from many projects, fatigue assessment is much more than collecting conventional Woehler lines, as especially industrial companies look for new time- and cost-effective testing approaches. WPT has patented the so-called "Rapid Fatigue Performance Identification Method (RAPID)" which works successfully for all investigated metals as well as polymer- and wood-based composites. The idea of RAPID is to estimate fatigue characteristics in short time for process-oriented manufacturing. RAPID allows to calculate the Woehler line on the basis of one multiple step test and two single step tests. The basics are presented in Walther, 2014, see below.

Picture 3: Prof. Walther (fifth from the right), Prof. Pogodaev (fourth from the right) and Shimadzu staff at Shimadzu fair, Vladivostok, Russia

Shimadzu:

Why are you interested in this research? What is the goal?

Prof. Walther:

Mechanical properties are combined with microstructure or microstructural changes due to production process and in-service loading. In this sense, microstructure could be understood as a fingerprint of the component properties and new non-destructive measuring techniques allow to describe the relation between microstructure and properties.

During my PhD, I worked on the fatigue assessment of ICE train wheels, as there was the Eschede (Germany) accident in 1998. My research topics are: quenched and tempered high strength steels for highly loaded parts, stainless steels for chemical plants, cast irons, lightweight metals as aluminium, magnesium and titanium, and recently carbon-fiber reinforced polymers (CFRP) and wood-based composites like vulcanized fiber. Additionally, joining techniques like friction-stir welding, brazing and bonding are investigated.

The goal is to understand the fatigue performance and failure mechanisms with the help of new measurement techniques on the basis of a small number of tests, to support industry in optimizing their process chain parameters regarding optimized component properties. The desired local component property should be reached on the basis of a well-understood structure-property relationship. Besides, the determination of the influence of corrosion on the fatigue properties and corrosion fatigue properties as well as corrosion prevention procedures are a further central research topic at WPT.

Shimadzu:

How are our instruments helping you?

Prof. Walther:

The instruments and the installed, Shimadzu as well as self-developed, softwares build the fundamental basis for experimental investigations (Picture 4). From WPT staff and many student workers, I know that especially the autograph software is very customized and self-explanatory. Excellent hardware and software quality are important criteria for the successful use of instruments to reach precise results in research and industry projects with minimum efforts.

Shimadzu:

What are Shimadzu's strengths compared to other competitor companies (not limited to the instruments) ?

Prof. Walther:

One of the Shimadzu strengths is to have a complete range of testing systems required for optimized investigations in research and industry. Besides the quality and capability of testing devices, I appreciate the close relationship to Shimadzu Europa headquarter. The European testing material team understands the demands of research institutes and supports us in a proper way. So we quickly learned that Shimadzu is treating their customers not only as a client, but as real partners. I am also deeply impressed about the high professional skills of the support team and the service engineers. The network serves as an ideal platform for information exchange and user discussions, also in the sense of new developments together with customers. Being comprehensive and communicative to its customers is the core competitive asset of Shimadzu.

Shimadzu:

Finally, could you share any requests that you have with respect to analytical and measuring instrument vendors?

Prof. Walther:

Actually, everybody in the fatigue community speaks about Very High Cycle Fatigue. In this year there is the 6th International Conference on Very High Cycle Fatigue (VHCF6) in Chengdu, China. I was very surprised when I heard that Shimadzu has a USF-2000 device in its portfolio. I am sure that this is not well-known in Germany, so marketing efforts and representation at prestigious conferences should be increased.

In case of lightweight alloys, the crash behavior is mandatory for industrial applications. Due to CE reasons, the HITS system, which I used in Kyoto for tests up to 20 m/s with a force up to 10 kN is unfortunately not available in German market.

I would recommend Shimadzu to proceed in the same way regarding quality of products and qualification of engineers, but to transfer more systems from Japan to Germany and increase marketing, especially for instruments needed in current research topics, and

conference attendances.

I am very happy that Shimadzu attends and supports, as Premium sponsor, the 1. Dortmunder Werkstoff-Forum from September 30 until October 1, 2014, in Dortmund, Germany, organized by myself.

Shimadzu:

It was significant to know what you think of us and our collaboration. We will strive to meet your expectations more than ever. Thank you very much.

Picture 4: Impressions from WPT labs, TU Dortmund University, Germany

Here are his recent publications:

- Wycisk, E.; Emmelmann, C.; Siddique, S.; Walther, F.: High Cycle Fatigue (HCF) performance of Ti-6Al-4V alloy processed by Selective Laser Melting. Advanced Materials Research 816-817 (2013) 134-139.
- Klein, M.; Wittke, P.; Dieringa, H.; Walther, F.: Influence of corrosion on fatigue properties of new creep-resistant magnesium alloy DieMag422. LCF 7, 7th Int. Conf. on Low Cycle Fatigue, Proc., ISBN 978-3-9814516-2-7 (2013) 319-324.
- Dieringa, H.; Huanga, Y.; Wittke, P.; Klein, M.; Walther, F.; Dikovits, M.; Poletti, C.: Compression-creep response of magnesium alloy DieMag422 containing barium compared with the commercial creep-resistant alloys AE42 and MRI230D. Materials Science and Engineering A 585 (2013) 430-438.
- Frieling, G.; Walther, F.: Tensile and fatigue properties of Fiber-Bragg-Grating (FBG) Sensors. Sensors & Transducers Journal 154, 7 (2013) 143-148.
- Manka, M.; Wojarski, L.; Tillmann, W.; Frieling, G.; Myslicki, S.; Walther, F.: Fatigue behavior of brazed AISI 304 joints using Au-fillers. Loet 2013, 10th Int. Conf. on Brazing, High Temperature Brazing and Diffusion Bonding, Aachen, DVS-Berichte Band 293, ISBN 978-3-87155-611-1 (2013) 232-236.
- Holweger, W.; Walther, F.; Loos, J.; Wolf, M.; Schreiber, J.; Dreher, W.; Kern, N.: Non-destructive subsurface damage monitoring in bearings failure mode using fractal dimension analysis. Industrial Lubrication and Tribology 64, 3 (2012), 132-137.
- Starke, P.; Walther, F.; Eifler, D.: "PHYBAL": a short-time procedure for a reliable fatigue-life calculation. Advanced Engineering Materials 12, 4 (2010) 276-282.
- Walther, F.: Microstructure-oriented fatigue assessment of construction materials and joints using short-time load increase procedure. MP Materials Testing 56, 7-8 (2014) 519-527.
- Walther, F.; Eifler, D.: Cyclic deformation behavior of steels and light-metal alloys. Materials Science and Engineering A 468-470 (2007) 259-266.
- Walther, F.; Eifler, D.: Fatigue life calculation of SAE 1050 and SAE 1065 steel under random loading. International Journal of Fatigue 29, 9-11 (2007) 1885-1892.

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Influence of Deformation Speed and Humidity on Quasistatic Deformation Behavior of Vulcanized Fiber – Experiments with Different Load and Climate Profiles.*

Bastian Peening, Daniel Hülsbusch, Frank Walther, Dominik Dumke and Bernd Künne, Dortmund

*Extended Abstract of paper published in MP Materials Testing in 2013, see bibliography in Chapter 5.

ABSTRACT

The detailed knowledge of the material properties is the basic requirement for design and production engineers, to ensure safe and efficient operation conditions of structural components. The insufficiently studied construction material vulcanized fiber shall be investigated microstructure-based and evaluated, regarding the influence of deformation speed and relative humidity on the quasistatic properties. Besides materialographic and microscopic investigations, an ultra-micro hardness tester was used. The mechanical characterization was done with an electromechanical universal testing system, a clip-gage and a video extensometer, thermocouples and a thermography system and a high-speed camera to detect crack formation and propagation.

1. Introduction

In consequence of stagnating research activities only a few material properties of vulcanized fiber can be found. Technical vulcanized fiber consists of cotton linters and/or recycled rags. Its fibers are processed to make absorbent and unsized special papers, which are joined by a merging process into one homogenous material by adding a parchmentizing solution. After bonding, the parchmentizing solution is leached out in a multistage process by using osmotic forces. Finally, the vulcanized fiber is dried (Fig. 1). The material properties of technical vulcanized fiber are comparable to those of engineering plastics, but it is fully based on natural resources. This leads to a hygroscopic behavior, which has to be considered in the following investigations.

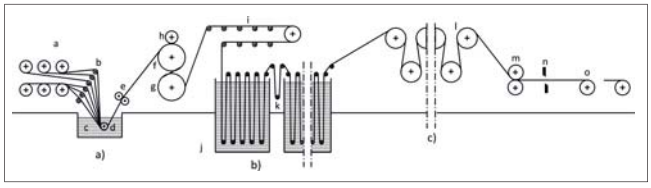


Fig. 1 Continuous production of vulcanized fiber

2. Experimental

2-1 setup and parameters

Specimens used in this investigation with the dimensions 140 mm x 15 mm were punched out of industrially manufactured vulcanized fiber with a thickness of 0.8 mm. From the wide field of influencing variables, deformation speed and relative humidity were chosen to determine their influence on the mechanical properties and the quantitative assessment. According to DIN 7738, the deformation speed has to be set to 10 % ± 2.5 % of the freely suspended length in mm/min. Speeds of 1, 5, 10, 100 and 1,000 mm/min were used in this investigation. The same DIN recommends climate conditions with 65 % relative humidity. In this case 25, 40 and 90 % relative humidity were taken into account. Therefore the specimens were conditioned in a climate chamber (Binder, Type KBF 240) at a temperature of 20° C. The tensile tests were performed directly after taking out the flat vulcanized fiber specimens of the climate chamber in a universal testing system (Shimadzu, Type AGS-X) with a maximum load of 5kN (Fig. 2).

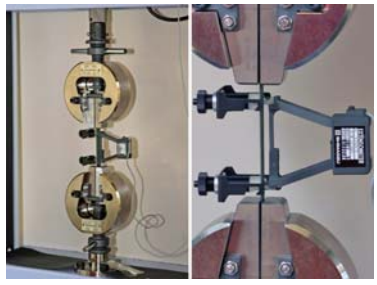


Fig. 2 Experimental set up for tensile tests

A precisely vertical clamping of the specimens with a freely suspended length of 70 mm excluded transverse forces. During the tests strain measurement was performed with an extensometer (Shimadzu, Type SSG25-50H) with a gage length of 50 mm.

2-2 results

Material - The aim of the production of vulcanized fiber is to dissolve the layer structure and merge the single fibers. This does not always succeed as Fig. 3 shows where the layers are differentiable from each other.

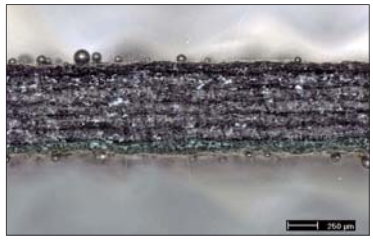


Fig. 3 Light micrograph, transversal cross-section

Scanning electron micrographs show the merging of the fibers in detail (Fig. 4).

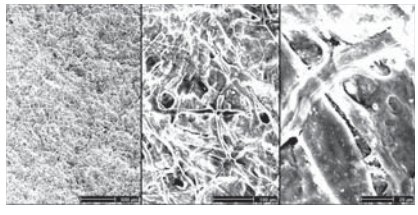


Fig. 4 Scanning electron micrographs (SEM)

Within the scope of the microstructural investigations, instrumented indentation tests according to DIN EN ISO 14577-1 were performed with an ultramicro hardness testing system (Shimadzu, Type DUH 211/S) as a load-unload-test. During the test the applied force in mN is logged as a function of the indentation depths in µm. The maximum force is held constant for a few seconds before relieving the load. After the test the elastic indentation modulus is measured by a tangent applied to the unloading curve and it corresponds to the E-modulus. For the tests a maximum force of 98 mN (10 P) was set according to HV 0.01.

Influence of deformation speed - For the investigations of the influence of deformation speed on the quasistatic material values, vulcanized fiber specimens were conditioned in 40 % relative humidity. Fig. 5 shows the strain hardening curves for 1mm/min and 1,000 mm/min deformation speed.

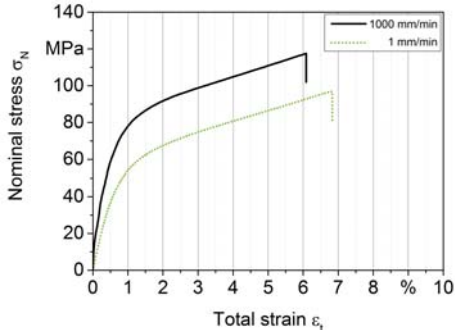


Fig. 5 Influence of the traverse speed on nominal stress-total strain-diagram

With an increase of the deformation speed from 1 to 1,000 mm/min, an increase of the E-modulus and the tensile strength and a decrease of the maximum strain can be seen. Fig. 6 shows the three material properties as a function of the deformation speed in linear or logarithmic scaling. At 1,000 mm/min the E-modulus reaches the highest value of nearly 16 GPa. In linear scaling the course of the curve indicates a saturation behavior so that deformation speeds > 1,000 mm/min are supposed to have only a small influence on this material property.

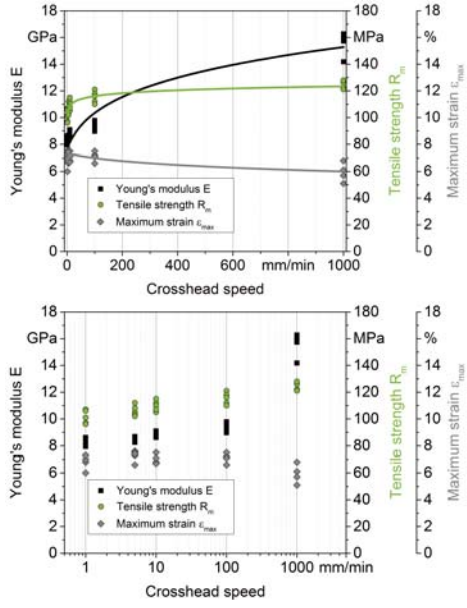


Fig. 6 Influence of traverse speed on E-modulus, tensile strength and maximum strain in linear and logarithmic illustration

Influence of relative humidity - Investigations have been performed at room temperature with a deformation speed of 10 mm/min. Fig. 7 shows the strain hardening curves for 25 % and 90 % relative humidity.

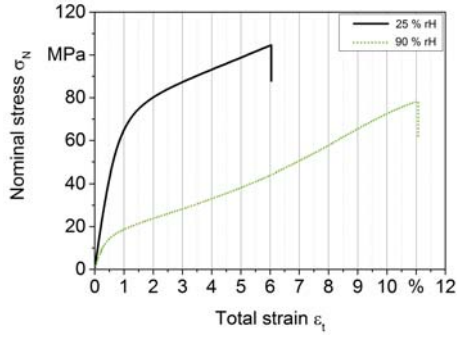


Fig. 7 Influence of the relative humidity on nominal stress-total strain-diagram

With rising relative humidity a decrease of the E-modulus and the tensile strength and an increase of the maximum strain can be seen. The countervailing effect of the curves is displayed in Fig. 8. As the abovementioned explanations show, both variables deformation speed and relative humidity have a strong influence on the material properties of vulcanized fiber.

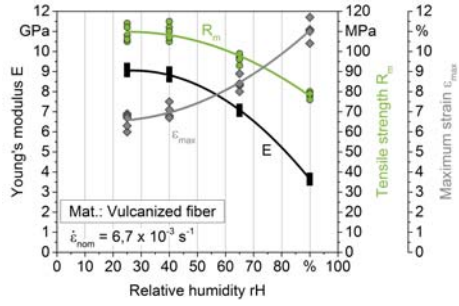


Fig. 8 Influence of relative humidity on E-modulus, tensile strength and maximum strain

Additional measurements - With the aim of a more detailed characterization of vulcanized fiber, further measurements have been used in the investigations. To exclude the damaging influence of the extensometer knifes a video extensometer (Shimadzu, Type TRViewX) has been used to measure the strain during the tests. Therefore reference marks have been attached to the specimen surface (Fig. 9). The results in strain measurement confirmed the measurements taken with the tactile extensometer.

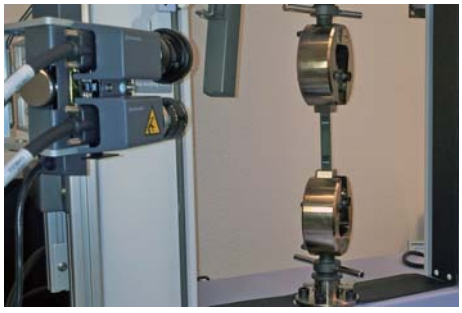


Fig. 9 Experimental set up with video extensometer

Further information about the material behavior was provided by thermometric measurements and infrared thermography. At the beginning of the test in the area of elastic deformation, the flat vulcanized fiber specimen absorbed energy in the form of heat which leads to a decrease in temperature. At the transition between the elastic and the elastic-plastic area, the temperature increased till the fracture of the specimen (Fig. 10).

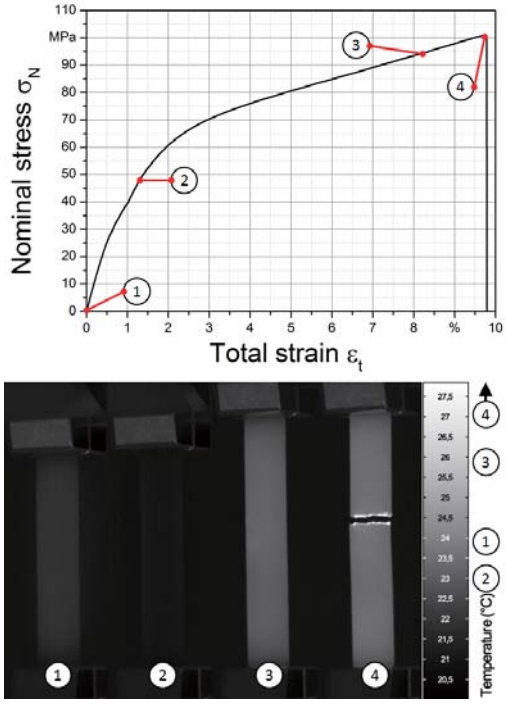


Fig. 10 Thermal images of different tensile test stages

Additionally the crack formation and the crack growth were analyzed. Thermography (Infra-Tec, Type IR8800) provided a reliable indicator of the starting point of cracks. A high-speed camera (Shimadzu, Type Hyper Vision II) was used for the investigation of the crack propagation (Fig. 11).

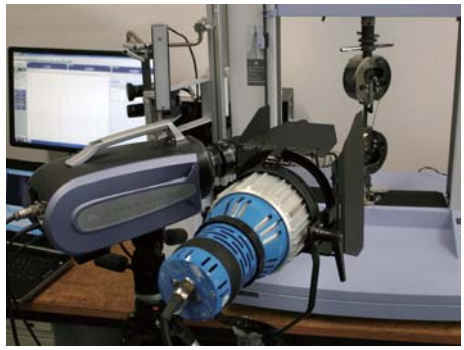


Fig. 11 Experimental set up with high-speed camera system

With a recording frequency of 500,000 frames per second, the time from crack formation to failure of the specimen could be determined with 3 x 10^{-4} seconds (Fig. 12).

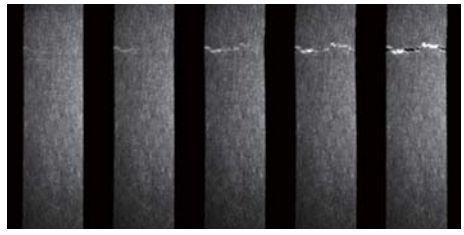


Fig. 12 Crack formation and propagation behavior in tensile test

3. Conclusions

For the characterization of technical vulcanized fiber with different load and climate profiles additional to conventional stress-strain-measurement, a video extensometer, a thermography system and a high-speed camera were used. Deformation speed and relative humidity have a significant influence on the quasistatic material properties. An increase in relative humidity leads to an increase in maximum strain and a decrease in E-modulus and the tensile strength. With an increasing deformation speed E-modulus increased too, but tensile strength and maximum strain showed a maximum value in the middle region. The use of additional measurement systems was well suitable for the detailed characterization of the deformation behavior of vulcanized fiber.

4. Outlook

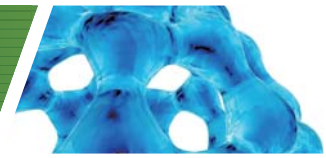
This article provides quasistatic values of technical vulcanized fiber. The scope of a following article is the characterization of the fatigue behavior. On the basis of quasistatic and cyclic material values it is possible to optimize the process- and product-oriented material properties. This leads to various new fields of application.

5. Extended Abstract of:

Penning, B.; Walther, F.; Dumke, D.; Künne, B.: Einfluss der Verformungsgeschwindigkeit und des Feuchtegehaltes auf das quasistatische Verformungsverhalten technischer Vulkanfiber. **MP Materials Testing 55, 4 (2013) 276-284.**

http://www.wpt-info.de

Adoption of a 10 MN Fatigue Testing Machine to Expand the Evaluation Ability of Composite Materials for Aerospace Structure



Sunao Sugimoto
Advanced Composite Research Center, Institute of Aeronautical Technology, Japan Aerospace Exploration Agency

In recent years, there has been a rapid increase in applications for carbon fiber reinforced plastics and other composite materials for commercial passenger aircraft. Even as much as half of the weight of some aircraft being used today consists of composite materials. When building aircraft, not only do strict safety requirements need to be met, but the need for lightness also should be satisfied. For these reasons, there is little leeway when it comes to safety as compared with other structures. The Composite Materials Handbook-17 (CMH-17) provides information and guidance necessary to design and fabricate aircraft structure from composite materials.

As a method for assuring the strength of composite material structures, CMH-17 offers the concept of a Building Block Approach (BBA), as shown in Fig. 1. In this approach, strength data for "coupons (strip-shaped samples)" is acquired first. Subsequently, the mechanical properties of "elements" such as C type and T type specimens are measured and then structural members "details" and "subcomponents" are measured. Finally, strength measurements for the target "components" are performed to produce the accumulated strength data. This method allows the strength of the target structure to be assured statistically.

Thousands of tests need to be performed like this because composite materials are inherently anisotropic and non-homogeneous. Compared with metallic materials, they exhibit multiple types of coexisting strengths. From a statistical point of view, there is an "A" value, which means that at a 95 % confidence level, 99 % or more of the test samples are strong enough to withstand destruction. There is also a "B" value, which means that at a 95 % confidence level, 90 % or more of the test samples are strong enough to withstand destruction. In the field of composite materials, the "B" value is most commonly used. In order to obtain the value for "B," n = 29 test samples are required for each item being tested. In many cases, this is rounded up to 30. Moreover, in the case of composite materials, there is a close connection between the manufacturing process and strength or inconsistencies in the strength. For this reason, five independent batches are commonly made. From each batch, six test samples are selected to form a total of 30 samples to be tested. With this, the uncertainties of the manufacturing process can be also evaluated.

At the Advanced Composite Research Center, Institute of Aeronautical Technology of JAXA, research aimed at expanding the applications for composite materials in aircraft, rockets, etc. is underway. So far, in research aimed at lowering the cost of composite materials for aircraft, we have implemented Vacuum-assisted Resin Transfer Molding (VaRTM) research for main wing box structures. As part of this, we have, as shown in Fig. 1, performed testing starting at the coupon level, proceeded onward to test full-scale simulations of the wing structure, and then performed finite element analysis corresponding to this. This follows the BBA. As one example, we have used a 2.5 MN hydraulic fatigue testing machine to perform a fatigue test on a large stiffened panel that simulates a portion of the lower skin panel of a main wing, and then have performed finite element analysis corresponding to this. Fig. 2 shows the results. In this fatigue test, as a result of the prior finite element analysis, we succeeded in very accurately predicting, within a 1 % margin of error, the load at which delamination will start at the end of the stringers.

As shown in Fig. 2, the need for the testing of large-scale samples that simulate aircraft structures has been felt both within and outside JAXA. In particular, in order to perform a compression test, a large-scale anti-buckling jig needs to be placed around the test sample, and in many cases, the space between the columns of the testing machine needs to be quite wide. Additionally, in the area of research and development for rocket structure, there is a need for load tests that can handle a portion of a full-scale structure. Moreover, due to the increasing size of test samples and the technologically advanced nature of materials, the need for a testing machine that is capable of handling much greater loads and provides a sufficiently wide amount of space between its columns is increasing.

At JAXA, however, the existing testing machine could only perform static tests up to 3 MN and fatigue tests up to 2.5 MN. The space between the columns of the testing machine was also narrow. Consequently, in February, 2014, we installed a Shimadzu Servopulser 10 MN fatigue testing machine (EHF-UV8MN-830). An overall view of this testing machine appears in the photograph in Fig. 3; Table 1 lists its specifications.

Immediately after adopting this 10 MN fatigue testing machine, it was used to test a 2.5 m dia. cylindrical shell with a composite lattice structure, which is a research model of a rocket structure in the Space Transportation Program by Space Transportation Mission Directorate, JAXA. Additionally, at the Advanced Composite Research Center, we are also proceeding with preparations for the compression strength testing of three stringer stiffened panels, as part of our research into low-cost composite materials.

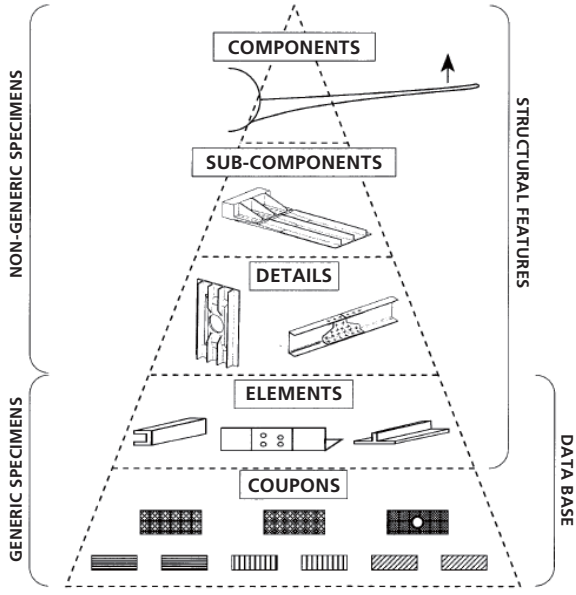


Fig. 1 Conceptual diagram of the building block approach (BBA)

Moreover, plans are being formulated for the implementation of a variety of tests of large structures, including tests of large-sized panels related to the development of commercial passenger aircraft, research and development related to eco-wing technology, which is being promoted by JAXA, and research and development into design technology for high-strain, lightweight, composite constructions. We expect this testing machine to play an even greater role in the testing of a variety of large-scale partial structures in the aerospace field.

As a final note, I would like to express my gratitude toward all the personnel from Shimadzu who were very helpful in getting this testing machine installed.

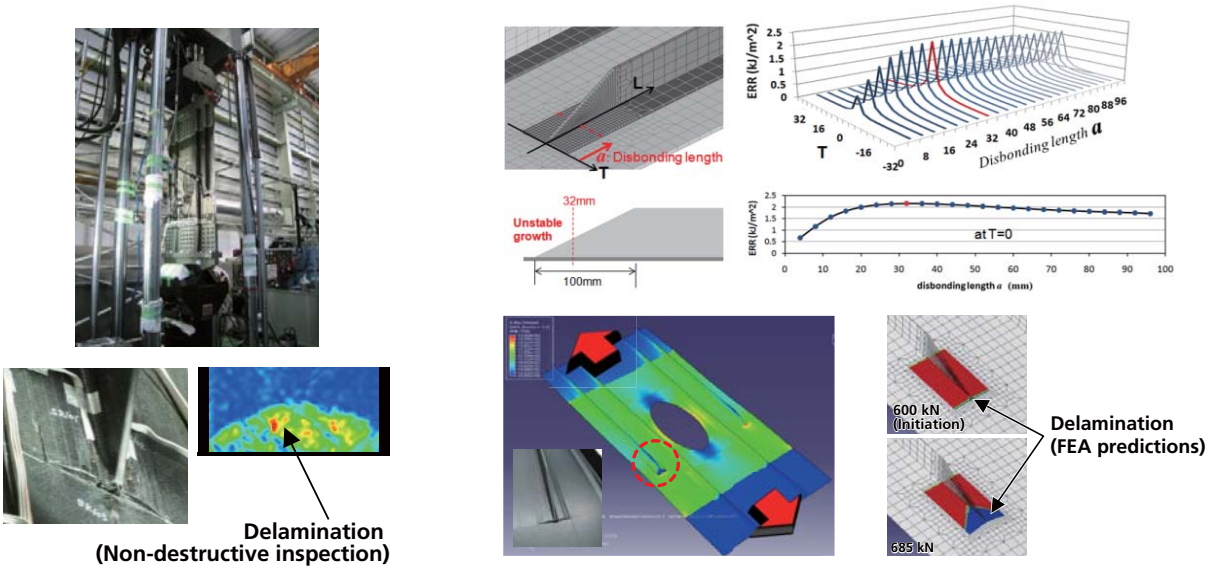


Fig. 2 Comparison between non-destructive Inspection result and finite element analysis prediction of VaRTM composite material stiffened panel under fatigue testing

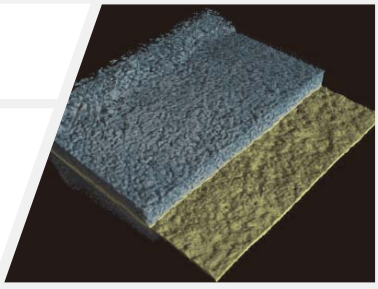
Fig. 3 Photograph of Shimadzu Servopulser 10 MN fatigue testing machine

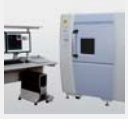
Table 1 Specifications of 10 MN Fatigue Testing Machine

Item	Specifications
Load Capacity	±10 MN static, ±8 MN fatigue
Actuator Stroke	±150 mm (Complete stroke: 300 mm)
Permissible Height of Load (distance between upper compression plate and table)	1000 mm to 4000 mm
Effective Load Space	3000 mm × 3000 mm

Shimadzu Selection

Shimadzu selected the following articles for this issue. They derive from application notes related to material sciences, and utilize a variety of instruments we produce. Cutting-edge technologies are also included.





Selection 1 Lithium-Ion Battery

X-Ray CT Observation of Lithium-Ion Battery Electrodes

Today, rechargeable lithium-ion batteries are widely used in a variety of fields and are available in a wide range of shapes, capacities, and applications. X-ray CT systems are able to non-destructively observe the internal structure of items. Therefore, they can be used to analyze defective batteries, compare conforming and nonconforming batteries, compare battery status before and after charging or discharging, evaluate changes in the internal structure of batteries during cycle testing, and so on.

Selection 2 Lithium-Ion Battery

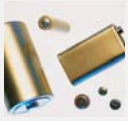
Lithium Ion Battery Binder Observations and Measurements of Physical Properties in Electrolyte Solution Using Scanning Probe Microscopy (SPM)

Lithium ion batteries are being developed for use in hybrid cars and electric vehicles, and further improvements are expected in terms of increased power output and battery performance. Lithium ion batteries are composed of a cathode, anode, separator and electrolyte. Normally the anode is made from a graphite active material, but recent years have seen research into silicon (Si) active materials as next-generation anode materials that will have a higher theoretical capacity than graphite active materials. Here, we carried out SPM shape observations of binder samples held in both the electrolyte material used for actual battery operation and in N2 gas for reference.

Selection 3 Lithium-Ion Battery

Simultaneous Analysis of Evolved Gas Produced by the Degradation of a Lithium-Ion Battery

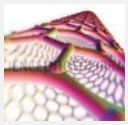
In evaluating the degradation of lithium-ion rechargeable batteries, it is necessary to analyze the gases produced inside the battery. The composition of the sampled internal gases can be investigated by conveying them to a gas chromatograph. The Shimadzu Tracera High-Sensitivity Gas Chromatograph uses a revolutionary plasma technology to detect all compounds except He and Ne. The system is capable of the simultaneous analysis of C1 to C3 hydrocarbons and inorganic gases including hydrogen, so it eliminates the conventional need for carrier gas switching or combined use of multiple systems. This Data Sheet introduces the simultaneous analysis of internal gases from a lithium-ion rechargeable battery utilizing the Tracera system.



Selection 4 Lithium-Ion Battery

Analysis of degradation products in electrolyte for rechargeable lithium-ion battery through high mass accuracy MS^n and multivariate statistical technique

The electrolyte of a LiB is consisting of a lithium salt in an aprotic organic solvent. The typical operational potential of a LiB is between 0 and 5 V. Therefore, solvent can be reduced or oxidized at the negative and positive electrodes during the battery charging process. As a result, various degradation products are generated in the electrolyte and cause some problems such as a decrease in the capacitance of battery. Here, we present the analysis method of degradation products generated in electrolyte using high mass accuracy MS^n and multivariate statistical technique.



Selection 5 Carbon Fiber Reiforced Plastic

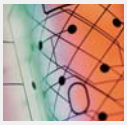
Analysis of Polyimide CFRP by TG-FTIR

Epoxy resin is typically used in carbon fiber composite materials (carbon fiber reinforced plastic: CFRP), however, due to its limited heat resistance, high heat-resistant CFRP materials using polyimide resin are being developed. Here, we introduce the results of combined Thermogravimetric (TG) - Fourier Transform Infrared Spectroscopy (FTIR) analysis of thermoplastic polyimide and thermosetting polyimide prepregs, polyimide matrices impregnated with carbon fibers and formed into sheets ready for processing.

Selection 6 Cement

Quantitative Analysis of Cement by EDX-8000

High accuracy quality control analysis of cement is typically conducted using a wavelength dispersive X-ray fluorescence spectrometer. EDX instruments have become capable in recent years of analysis accuracy comparable to that of low-output wavelength dispersive type instruments. Not only do they offer such conveniences as the ability to analyze powders as they are, their range of applicability has greatly widened to include the analysis of such substances as cement. Here, we introduce the analysis accuracy obtained with pressure-formed cement standard powder samples using the new model EDX-8000.



Selection 7 Polymer Film

Mechanical Strength Evaluation of Functional Film Used in Smartphones

Plastic film is formed into thin film using techniques such as extrusion and stretch molding of the polymer film. A wide variety of products having special character ist ics, such as water retentivity, light reflectivity, and selective permeability for specific substances, can be found all around us as industrial products, construction materials, as well as everyday necessities.

Selection 8 Perfumes

GC/MS Quantitative Analysis of Allergens in Cosmetics Using OPTIC-4

Because an estimated 1 to 2 percent of the population has allergies, in 2003 the EU began regulating the use of perfumes and flavor materials that can cause allergic reactions. These substances are restricted to 0.01 % for cosmetic products that are rinsed away (such as shampoo) and 0.001 % for products that are left applied (such as hand creams). Due to these regulations, manufacturers now must quantitatively analyze the concentration of such allergens.

Selection 9 Surface Analysis

Development of Spheroid Energy Analyzers for Elemental and Chemical Analysis of Surfaces

The spheroid energy analyzers (SEA) are a new class of charged particle energy analyzers that are characterized with very high energy resolution and transmission. The design of the first prototype analyzer is described. A novel geometrical framework is presented, that describes SEA analyzers in general terms within which the well known types of analyzers CMA and CHA appear to be only particular examples. A very high order of focusing of these analyzers is presented via simulation that indicates the existence of 13th order focusing in one of our models. Experimental results are presented showing relative energy resolution of 0.05% of the pass energy at a transmission of 21% out of a 2π steradian.

Selection 10 Elemental Analysis

X-ray Fluorescence Analysis of Lead in Tin Plating Using Theoretical Intensity of Scattered X-rays - Analysis of RoHS Regulated Elements by Energy Dispersive X-ray Fluorescence Spectrometer (EDX) -

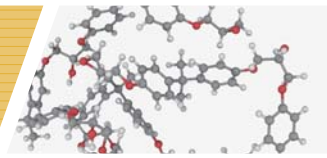
RoHS regulated elements, such as cadmium and lead, are analyzed by energy dispersive X-ray fluorescence spectrometer (EDX). However, the quantitative values obtained by this method are affected by the thickness when the sample is a thin film, such as plating. Therefore, we measured the film thickness in an attempt to correct the quantitative values.

Selection 11 New Technology

Development of Transparent Encapsulation Films for OLEDs Using Surface Wave Plasma CVD System

We have succeeded in developing a highly transparent, water impermeable SiNx encapsulation films for organic light-emitting displays using a surface-wave-plasma enhanced chemical vapor deposition (SWP-CVD) system. A highly reliable barrier performance (no deterioration for more than five years under the room temperature) was realized with above SiNx film as an encapsulation of OLEDs.

Analysis of Minor Components in Synthetic Polymers Using SEC-AccuSpot-AXIMA System



MALDI-TOF mass spectrometry is widely used as a technique for the characterization of synthetic polymers. However, when multiple components are present in samples, major components may suppress ionization of the trace components present, preventing their detection. Size exclusion chromatography, (SEC) is a chromatographic method that can be used to prevent this adverse result by separating the components into fractions beforehand. However, off-line SEC/MALDI MS involves fraction collection, evaporation, pipetting, etc. and is laborious and time consuming, so direct deposition methods in which SEC fractions and MALDI matrix are directly deposited onto the MALDI target are preferred. Shimadzu has developed the AccuSpot automated spotter to mix the eluate from the LC with the various reagents and directly load them onto the MALDI sample plate. As a result, an automated analytical SEC-MALDI analysis system was constructed that can rapidly simplify all the processing from LC separation to MALDI-TOFMS measurement. Here, using a copolymer - poly (methylmethacrylateb-n-butylmethacrylate) (poly (MMA-b-n-BMA)) -a sample with a complicated composition, we present an example of detection of a homopolymer present at trace levels in the sample. The size exclusion chromatography (SEC) mode was used for the separation, and a micro-scale separation column was used to load all of the components onto the MALDI sample plate without any waste. The AccuSpot body is constructed with enhanced resistance to many organic solvents used in the field of synthetic polymers.

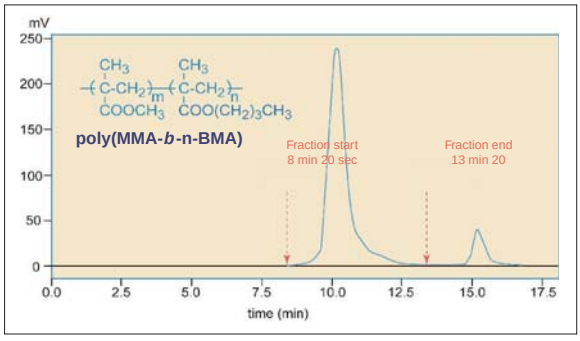


Fig. 1 SEC Chromatogram of Poly (MMA-*b*-n-BMA)

Table 1 Analytical Conditions

• SEC	Column	: Shodex GF310A-1E (1.0 mmI.D.× 250 mm)
	Flow Rate	: 10 µL/min, Eluent : THF
	Detector	: UV (λ=220 nm), Injection volume : 1µL
• AccuSpot	Spot Interval	: 6 sec, Loadage : 1µL/well
	Loadage (mixture of matrix and cationizing reagent)	: 0.2 µL/well
• MALDI-TOFMS	Matrix	: Dithranol-20 mg/mL- THF
	Cationizing Reagent	: Na-TFA-10 mg/mL -THF

THF : Tetrahydrofuran, TFA : Trifluoroacetic acid

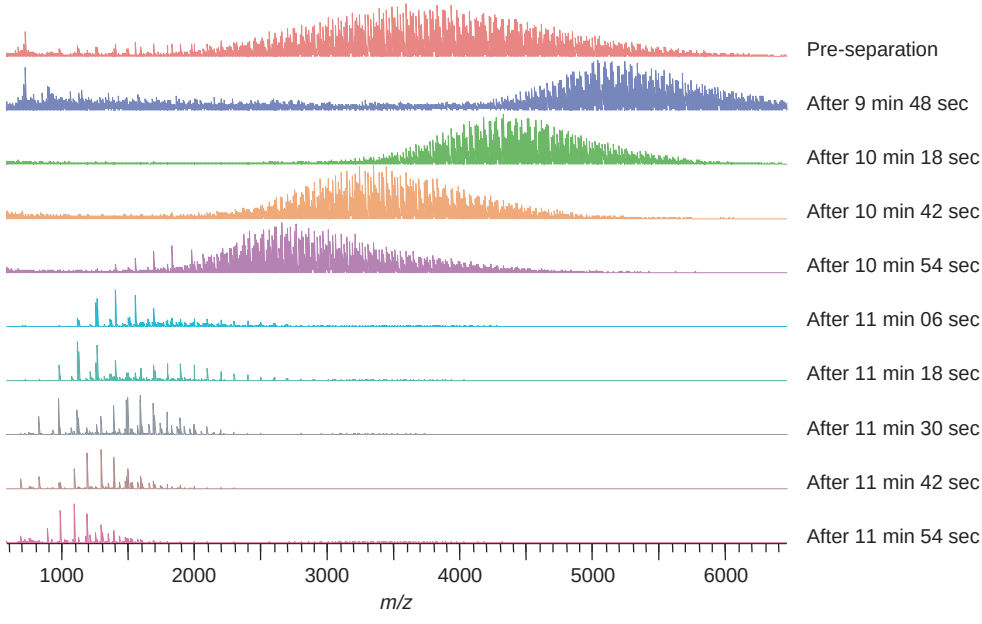


Fig. 2 MS Spectra of SEC-Separated Fractions

Fig. 1 shows the SEC chromatogram of poly (MMA-*b*-n-BMA). The main peak was collected every 6 seconds from the start to the end of elution (8 min 20 sec to 13 min 20 sec) using the AccuSpot, and 50 sample spots were created on the MALDI sample plate. Each spot was measured using the MALDITOFMS instrument, and the mass spectra corresponding to the elution times were obtained (Fig. 2). The mass spectra obtained from the fractions each show a different molecular weight distribution. Focusing on the fraction after 11 min 18 sec, two different types of molecular weight distribution were detected that had not been seen before separation (Fig. 3). The peaks indicated by● in the molecular weight distribution with *m/z* 1200 at the top show an interval of 142 between adjacent peaks, a distribution indicating the existence of the PnBMA homopolymer. On the other hand, the peaks indicated by ■ in the molecular weight distribution with *m/z* 1800 at the top show an interval of 100 between adjacent peaks, indicating the existence of the PMMA homopolymer. The above results clearly show that the PnBMA and PMMA homopolymers are present as minor constituents in the poly (MMA-*b*-n-BMA) sample used here. The detection of these minor components included in the synthetic polymer demonstrates that this SEC-AccuSpot-AXIMA System achieves higher time resolution of eluted constituents than the manual SEC/MALDI-TOFMS technique previously (Fig. 4).

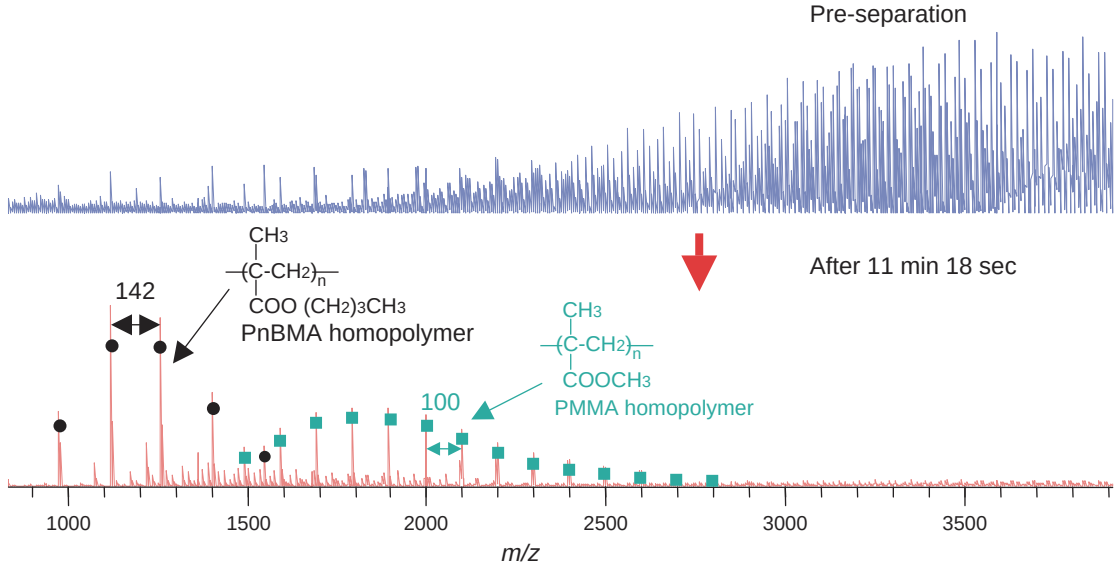


Fig. 3 Detection of Homopolymer Components

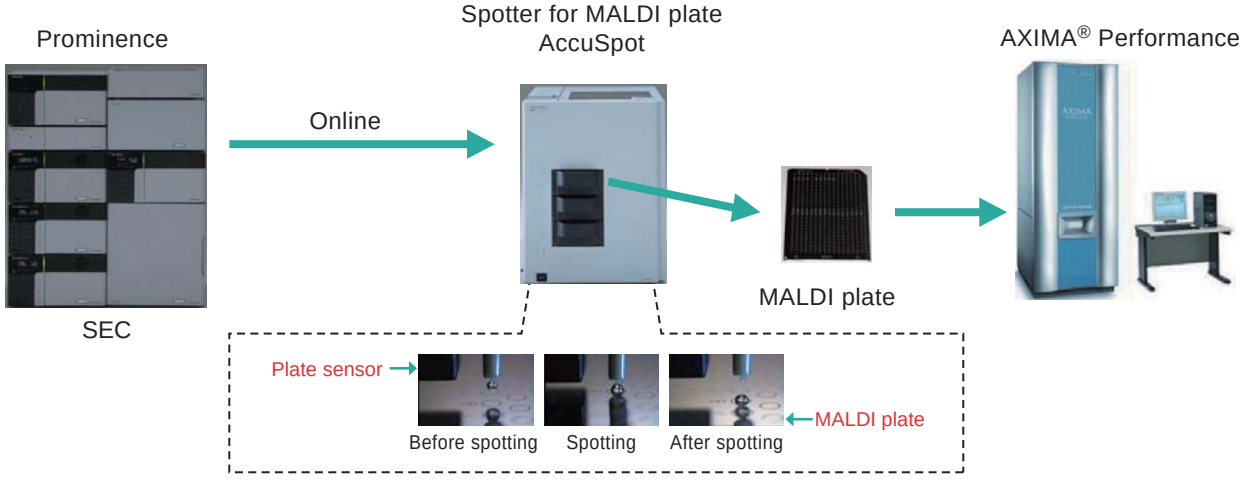


Fig. 4 SEC-MALDI System

NOTES:
✻ This Application News has been produced and edited using information that was available when the data was acquired for each article.
This Application News is subject to revision without prior notice.

Shimadzu Global Laboratories Network

Japan---"Global Application Development Center" at Headquarters

The Global Application Development Center in Japan plays the most significant role as Shimadzu's leading state-of-the-art laboratory. It provides a total solution that includes instrument evaluation, analytical consultation, and customer training programs. In this laboratory, Shimadzu scientists and engineers continually develop new analytical methods and novel applications to meet customer requirements.

The United States--- "Solution Center"

Shimadzu Scientific Instruments (SSI), US, has opened its Solution Center at its Columbia, Md. headquarters in order to enhance collaboration with customers and to enable the company to quickly respond to customer needs for new scientific instruments, software platforms and applications. SSI held an opening ceremony on March 12, 2014. A US Congressman, senior state government officials, and professors from distinguished universities attended the ceremony to celebrate event.

The Solution Center showcases more than 30 scientific instruments highlighting SSI's full range of products and capabilities, including molecular/atomic spectrophotometers, mass spectrometers, liquid/gas chromatographs, X-ray spectrometers, balances, TOC analyzers, and materials testers. The center also features high-definition state-of-the-art video and conferencing equipment to facilitate customer interactions both within the center and in collaboration with manufacturing, software, and R&D groups.

Europe---"Laboratory World"

Shimadzu Europe GmbH (SEG) has created a new Laboratory World at its Duisburg, Germany headquarters to serve its customers, who come from all over Europe. Laboratory World is spread across an area greater than 1,500 square meters (m^2) and features testing facilities for Shimadzu's entire product line, ranging from chromatographs, spectrophotometers, TOC analyzers, mass spectrometers and balances to materials testing machines. Mass spectrometry, a technology that Shimadzu has significantly shaped in recent years, will be highlighted in a dedicated space. In addition, laboratory areas for customer applications and seminar facilities are also being expanded.

Shimadzu boasts several customer support and application development centers around the world, including three newly opened ones in the USA, Germany, and Taiwan.

China--- "Analysis Center"

There are five analysis centers in mainland China: Shanghai, Beijing, Guangzhou, Shenyang, and Chengdu. These function to develop solutions and to provide training programs with customers. Both the Shanghai and Beijing Analysis Centers have over a hundred sets of the latest analytical instruments located in 600 square meters (m^2) of laboratory space and about 200 square meters (m^2) of training facilities. Moreover, China National Accreditation Board for Conformity Assessment (CNAS) has recognized the Shanghai Analysis Center with a quality management system since March 2011. Furthermore, most centers are situated in central business districts, making them easily accessible by public transportation.

Taiwan---"Application Laboratory"

Shimadzu Scientific Instruments (Taiwan) Co., Ltd. (SST) was established on November 20, 2013 and features an application laboratory equipped with various analytical and measuring instruments, such as mass spectrometers and high-performance liquid chromatographs. This laboratory will be utilized to provide solutions to customers' needs for R&D or quality control work, and to provide customers with enhanced support.

Singapore---"Customer Support Center"

Singapore-based Shimadzu (Asia Pacific)'s Customer Support Center (CSC), which is the largest in Asia, is well-equipped with a state-of-the-art laboratory. It is furnished with a wide range of fully functional analytical instruments, dedicated training rooms and preparative laboratories for sample pre-treatment work. A team of highly-skilled application chemists focus on specific customer needs through training sessions focused on the operation and maintenance of the instruments, as well as method development. The CSC trains over 1,000 chemists annually.

India---"Customer Support Center"

Continuing Shimadzu's commitment to supporting its customers in India, the Customer Support Center (CSC) was established in Mumbai in 2006. The center operates in line with Shimadzu's inherent value - we are dedicated to being the "Best for Our Customers". The CSC lab is an absolute reflection of Shimadzu's capability of providing all-around solutions for any analytical lab's requirements. The lab extends support to all its customers in the form of hardware, application and software trouble-shooting and maintenance training. This ensures smooth operations, maximizes up-time, facilitates faster analytical development and, most importantly, enables better understanding of systems and technology.

Shimadzu (Asia Pacific) signs MoU with NERI, sets up NUS-Shimadzu Advanced Facility for Ecoanalytics (NUSAFE)

Singapore-based Shimadzu (Asia Pacific) and National University of Singapore Environmental Research Institute (NERI) have signed a Memorandum of Understanding (MoU) to establish a joint facility to conduct environmental analytical research. The facility will be called NUS-Shimadzu Advanced Facility for Ecoanalytics or NUSAFE.

Singapore, 2 June 2014 – The National University of Singapore (NUS) Environmental Research Institute (NERI) and Shimadzu (Asia Pacific) Pte Ltd, a global leader in the manufacturing of analytical instruments, testing instruments, precision instruments, measuring instruments and medical equipment, are collaborating to conduct research on environmental contaminants.

To mark this partnership, Mr Yasuo Miura, Director, Shimadzu Corporation, and Professor Ong Choon Nam, Director, NERI, signed a Memorandum of Understanding (MoU) on 2 June 2014. The collaboration will enable the two organisations to mutually benefit from each other's substantial expertise in the environmental sciences. A joint facility called the NUS-Shimadzu Advanced Facility for Ecoanalytics (NUSAFE) will also be established as part of the agreement.

Shimadzu will contribute instruments, such as a Mass Spectrometric (MS) system that can be used for microorganism analysis and identification, a Total Organic Carbon (TOC) analyser, and a Triple-Quadrupole Gas Chromatography Mass Spectrometer (GCMS-TQ), used for trace analysis of environmental contaminants, during the initial phase. These instruments allow for quick identification and quantitation of volatile metabolites and microorganisms in various environmental samples, which is key in investigating and understanding the relationship between these metabolites, microorganisms and the environment.

Additionally, NERI and Shimadzu will develop and apply fully automated and solvent-minimised sample preparation procedures that have been integrated with mass spectrometry for water quality monitoring. This analytical platform can be operated on site with minimal human intervention.

Mr Kiminobu Imura, Managing Director, Shimadzu (Asia Pacific) Pte Ltd, said, "It is a pleasure for a global organisation like Shimadzu to collaborate with a world-renowned institute like NERI. Being a global leader in analytical instruments impresses upon us the responsibility to provide the best infrastructure to the scientific community. The signing of this MoU has once again reiterated our commitment towards research in accordance with our Corporate Philosophy of "Contributing to Society Through Science and Technology." Excellence in Science is not just our motto; it is also a way of life for us at Shimadzu and we try to assimilate this value in this MoU."

Mr Prem Anand, Senior General Manager, Shimadzu (Asia Pacific) Pte Ltd, said, "We are elated to join hands with NERI in setting up the state-of-the-art NUS-Shimadzu Advanced Facility for Ecoanalytics (NUSAFE). The signing of this MoU will further enhance the capabilities of NUS-NERI, which is already well equipped with several advanced Shimadzu instruments. Investing in NUSAFE is a long-term and sustainable investment for Shimadzu. As a global leader in analytical instruments, we feel that it is among our foremost Corporate Social Responsibility to aid researchers and equip them with the latest technology, such as Ultra Fast Mass Spectrometry (UFMS)."

Professor Ong Choon Nam, Director of NUS Environmental Research Institute (NERI), said, "NERI is very pleased to have Shimadzu, one of the world's leaders in analytical instruments, as one of our research partners. The establishment of this joint facility, NUSAFE, will further enhance our laboratory's analytical capability and advance our environmental research. The partnership will also foster stronger ties and facilitate exchange of knowledge and research findings between academia and industry to enable both parties to stay competitive and relevant in their respective fields."

Signing of MoU between Shimadzu (Asia Pacific) and NUS at Singapore International Water Week: (From Left to Right) Ms Janice Chia, Sales Manager, SGP Sales, Shimadzu (Asia Pacific) Pte Ltd, Singapore; Mr Kiminobu Imura, Managing Director, Shimadzu (Asia Pacific) Pte Ltd, Singapore; Mr Yasuo Miura, Executive Director, Shimadzu Corporation, Japan; Professor Ong Choon Nam, Director, NUS Environmental Research Institute; and Professor Lee Hian Kee, Department of Chemistry, NUS, Singapore

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About Shimadzu (Asia Pacific) Pte Ltd

Shimadzu (Asia Pacific) is the Asian subsidiary of Shimadzu Corporation, Japan. The subsidiary was established in 1989 in Singapore as a distribution centre providing analytical solutions, scientific equipment, testing machines, balances and medical equipment to a wide range of laboratories in Asia. A fast paced growth of business in the region has seen the opening of 3 subsidiaries in India, Malaysia and the Philippines with application specialists, technical and sales engineers situated throughout the region. Additionally, the company operates via a wide and carefully selected network of local distributers in every region. The company's instruments are relied upon by researchers all across Asia from large multinational organisations to local academic research universities and a long list of local, state, and central laboratories. Through adoption of scientific inventions and cutting-edge technologies, Shimadzu (Asia Pacific) provides its corporate clients with the highest quality in product standards and the delivery of excellent customer support services.

For more information, please visit www.shimadzu.com.sg.

About National University of Singapore (NUS)

A leading global university centred in Asia, the National University of Singapore (NUS) is Singapore's flagship university, which offers a global approach to education and research, with a focus on Asian perspectives and expertise. NUS has 16 faculties and schools across three campuses. Its transformative education includes a broadbased curriculum underscored by multi-disciplinary courses and cross-faculty enrichment. Over 37,000 students from 100 countries enrich the community with their diverse social and cultural perspectives. NUS has three Research Centres of Excellence (RCE) and 23 university-level research institutes and centres. It is also a partner in Singapore's fifth RCE. NUS shares a close affiliation with 16 national-level research institutes and centres. Research activities are strategic and robust, and NUS is well-known for its research strengths in engineering, life sciences and biomedicine, social sciences and natural sciences. It also strives to create a supportive and innovative environment to promote creative enterprise within its community.

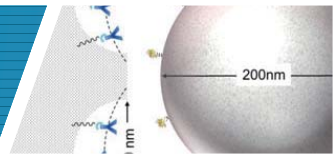
For more information, please visit www.nus.edu.sg

About NUS Environmental Research Institute

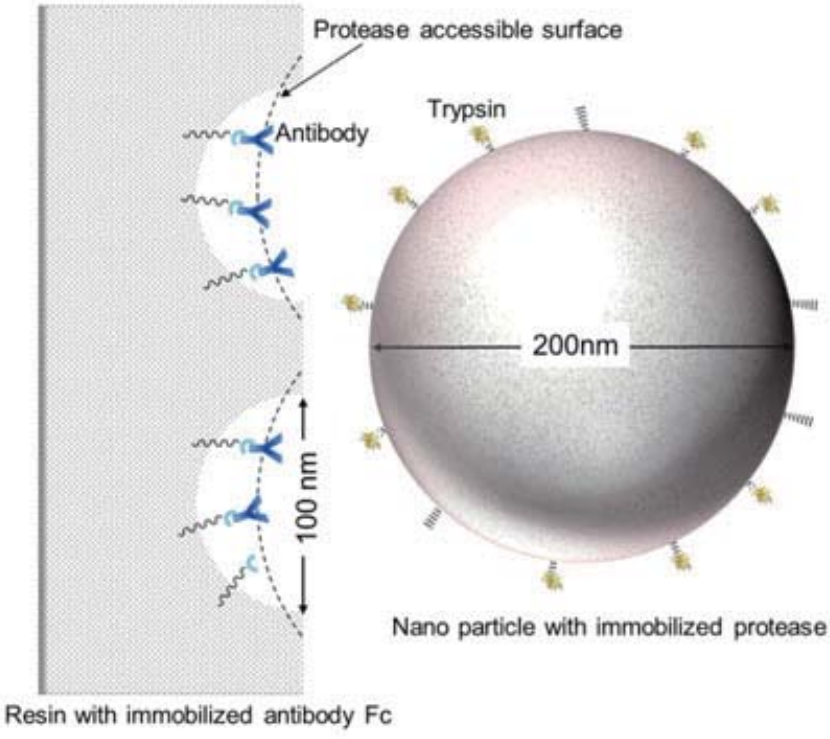
The National University of Singapore Environmental Research Institute (NERI) is a leading interdisciplinary centre for environmental research in Asia. The Institute brings together leading researchers and expertise across the NUS campus to conduct and foster collaborative, multidisciplinary environmental research; coordinate the development of strategic thematic research; train graduate research students for academic careers in the environmental sciences and engineering, and facilitate the transfer of technology to industry. A key thrust of NERI is to conduct innovative globally impactful research on scarce resources. Some of the current research undertaken within the Institute includes environmental monitoring, surveillance, and management of environmental pollutants, environmental and public health issues related to climate change, food security and safety, Green Chemistry and Sustainable Energy such as waste-to-energy conversion.

For more information, please visit www.nus.edu.sg/neri

Our paper of monoclonal antibody analysis by MS selected as Hot Articles



Our paper, Noriko Iwamoto et al., Life Science Research Center, Shimadzu Corporation, has been selected to the Hot Articles of Analyst in Royal Society of Chemistry. This report is one of the collaboration results of National Cancer Center in Japan. Our new method, named nSMOL proteolysis (nano-surface and molecular orientation limited proteolysis), is the innovative proposal to the antibody analysis by mass spectrometry focused on the size of antibody and its orientation. Immobilized antibody on the resin with 100 nm pore has been digested by immobilized protease on 200 nm nano particles, and detected and quantified by liquid chromatography/mass spectrometry (LC/MS). This experimental design successfully performed limited proteolysis on complementarity-determining region of the antibody variable region by limiting protease access to the substrate. This nSMOL method is made possible therapeutic drug monitoring (TDM) independent of a variety of monoclonal antibodies, and accelerated the development of TDM system with MS.



Selective detection of complementarity-determining regions of monoclonal antibody by limiting protease access to the substrate: nano-surface and molecular-orientation limited proteolysis

Noriko Iwamoto, Takashi Shimada, Yukari Umino, Chikage Aoki, Yutaka Aoki, Taka-Aki Sato, Akinobu Hamada and Hitoshi Nakagama
Analyst, 2014, 139, 576-580

http://blogs.rsc.org/an/2014/02/07/hot-articles-in-analyst-32/
http://pubs.rsc.org/en/Content/ArticleLanding/2014/AN/c3an02104a#!divAbstract

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