

# MATERIALS SCIENCE AND TECHNOLOGY NEWSLETTER

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## SURFACE SCIENCE, SILANES AND ADHESION

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## EDITORIAL COMMENTS

In addition to announcing the latest MST symposia and short courses which will be coming up within the next 12 months, this issue of the newsletter also announces a new feature to be introduced to the MST web site and a new blog being published in cooperation with the Plasmamatreat company.

The first symposium to be announced is the upcoming NINTH INTERNATIONAL SYMPOSIUM ON SILANES AND OTHER COUPLING AGENTS to be held at the Hampton Inn, Harriman-Woodbury, New York, October 3-4, 2013. This symposium continues the tradition set by the first symposium in this series: "Silanes and Other Coupling Agents" which was hosted in 1991 by the Dow Corning Corporation in honor of Dr. Edwin P. Plueddemann. As with its predecessors, this symposium will be concerned with the technological areas where the use of surface primers such as silanes are critical to the success of many technologies.

Note that the silane symposium will be held in conjunction with the short course on the CHEMISTRY, PHYSICS AND MECHANICS OF ADHESION SCIENCE which will be held from September 30 to October 2, 2013. Further details and registration information are given at the end of the newsletter.

Readers should also mark their calendars for the upcoming NINTH INTERNATIONAL SYMPOSIUM ON CONTACT ANGLE, WETTABILITY AND ADHESION to be held at Lehigh University, Bethlehem Pennsylvania, June 16-18, 2014. The contact angle measurement method has become increasingly popular in recent years due to a number of factors. Quite likely the leading one is the fact that the contact angle technique is likely the least expensive of all surface analysis methods with serviceable equipment going for under \$20,000. Another point is that, among the repertoire of surface analysis techniques, the contact angle approach is the only one that samples just the very top layer of the surface under investigation. In addition to these properties the contact angle method has become one of the leading tools in investigating several phenomena of current interest and importance including electrowetting behavior, superhydrophobic surfaces and self cleaning surfaces to name just a few. These and many more leading edge topics will be discussed at the Lehigh meeting.

We would also like to announce the inauguration of a new blog in cooperation with the Plasmamatreat Corp. This blog will discuss a range of surface phenomena dealing with surface modification and a number of other surface related topics. We

encourage readers of the newsletter to check out the blog web site which can be conveniently accessed at:

<http://www.plasmablog.com/>

Finally we want to bring attention to an upcoming feature on the MST web site whereby users will be able to carry out useful fracture mechanics calculations of surface coatings using their tablets and smartphones. More on this and other topics below.

## SURFACE SCIENCE CORNER

As mentioned above we have started a blog in cooperation with the Plasmamatreat company that will deal with a wide range of surface related phenomena. Plasmamatreat is a world wide supplier of atmospheric plasma equipment used in a wide range of industries to clean and modify polymer surfaces prior to coating. Needless to say the success of any surface modification treatment relies on having an understanding of surface properties thus the blog site is an integral part of further developing and supporting this activity.

Going under the title of SURFACE SCIENCE CORNER the topics to be covered are:

**ADHESION:** One of the primary uses of plasma technology is to alter the chemistry of a surface in such a way as to increase the surface energy and thus significantly improve the adhesion of candidate coatings. Plasma technology has been very effective in this regard. However, it is also important to know whether the level of adhesion achieved is sufficient for the purpose at hand so the topic of adhesion measurement will also be an important consideration.

**SURFACE CLEANING:** Surface cleaning is another important application of plasma technology and it is also critical to ascertain the level of cleaning attained in order to know whether it is sufficient or not. Thus measuring surface cleanliness is a further important issue.

**SURFACE ANALYTICAL METHODS:** The ability to analyze the chemical and physical nature of a surface is of course critical to both of the above mentioned topics. Thus attention will be paid to the various analytical methods used to investigate surface chemistry such as X-ray Photoelectron Spectroscopy (XPS also going under the alias Electron Spectroscopy for Chemical Analysis, ESCA). The thermodynamic nature of surfaces is effectively investigated through the use of CONTACT ANGLE measurements. This rather prosaic measurement technique, though simple in

concept, is quite subtle in practice and can provide a wealth of valuable information on the physico-chemical nature of surfaces.

**BOOK REVIEWS:** Our office receives a number of volumes dealing with all aspects of surface science. From time to time when a particularly relevant volume is received it will be brought to the attention of readers of this blog by way of a critical review.

**REVIEWS OF IMPORTANT CURRENT RESEARCH:** By way of keeping up to date on all aspects of surface science we organize each year through MST CONFERENCES up to 4 symposia which cover the latest developments in this field. From time to time a particularly relevant presentation will be given and such will also be brought to the attention of readers again through a critical review.

Finally let us conclude with a few remarks concerning the style and presentation level of this blog series. Our basic aim will be to make what is arguably a rather esoteric topic clear and understandable to the non-expert by giving concrete examples and also by avoiding technical jargon to the greatest extent possible. Thus we aim for what might be construed as a SURFACE SCIENCE FOR DUMMIES writing style. The aim, therefore, will be to make surface science topics clear and understandable to a range of individuals who have a need to know this topic but may not have either the background or time to pursue the matter at the level of the technical literature. A famous professor once told a student that if he could explain his work to a barmaid then he indeed knew what he was talking about. We will strive to achieve that level of clarity to the greatest extent possible.

Starting in August 2013 we start a series of discussions under the title:

### [SURFACES: THE INVISIBLE UNIVERSE WE LOOK UPON EVERY DAY BUT NEVER REALLY SEE](#)

This series of essays will deal with the general topic of the hidden aspects of surfaces that, though generally invisible to us, have an important influence not only on our day to day activities but also on our ability to manufacture common objects of everyday use. A typical example is the polymer materials we use to encapsulate all manner of foodstuffs to protect them from moisture, oxidation and other unwanted atmospheric influences. A typical packaging resin may be quite effective at protecting against unwanted atmospheric invaders but also impossible to label or decorate with

commonly available inks. The ability of a given surface to be written on by a given ink is controlled by the surface energy of the polymer and the surface energy of any given material is just one among a host of invisible surface properties. Thus the underlying thread in all of the discussion will be the fact that although most properties of surfaces may be invisible to us they nonetheless have a rather profound influence on not only our ability to manufacture items of common commerce but our everyday experience as well.

The following is an outline of the topics to be explored in more detail in subsequent issues of the blog:

1. **OPTICAL PROPERTIES:** What we see is only an infinitesimal fraction of the radiation emitted by surfaces.
2. **SURFACE ENERGY:** A world that can only be explored through the lens of thermodynamics.
3. **SURFACE ANALYSIS METHODS:** ESCA, AUGER, TOFF SIMS, EELS, ... the alphabet soup of surface analysis techniques that allow us examine surfaces at the atomic level.
4. **CONTACT ANGLE:** A poor man's surface analysis tool you can implement in your kitchen and curiously enough turns out to be superior to the high power methods in an interesting way.
5. **SURFACE FORCES:** Ever wonder why insects and the gecko lizard can walk upside down on your ceiling or nearly any other surface? The surface van der Waals forces are of exceedingly short range but their influence extends to surprisingly large distances.
6. **ADHESION:** Mostly you find that when two objects come into contact they do not adhere very well. Some materials, however, seem to stick to nearly everything. These behaviors can only be sorted out through an understanding of surface forces and interactions.
7. **CONTAMINATION:** Surfaces are an invisible refuge for all manner of foreign species and these invaders can alter the surface's properties in ways both benign and malign. The oil layer on the cylinder walls of your car's engine is absolutely critical to it's life and function. That same oil can also prevent you from painting over a bare spot on the fender.

8. TRIBOLOGY AND FRICTION: An invisible property that determines whether things stick or slip or whether your tires will keep you on the road or send you to the gutter.
9. SURFACE MODIFICATION: Getting surfaces to behave the way we want.
10. LIFE ON SURFACES: Microbes are another invisible inhabitant in the world of surfaces and have many important consequences including biofouling of pipes and marine surfaces and also the common infections that can lay us low.

Readers interested in industrial applications of surface modification and cleaning will find several other blogs on the Plasmatreat blog site dealing with issues related to microelectronics, aerospace, life sciences, packaging and polymer structure and thin films. The place to go is:

<http://www.plasmablog.com/>

Finally, we would like to thank Andy Stecker President Plasmatreat USA for giving us the opportunity to contribute to the Plasmatreat blog and we hope all readers will find topics of interest and enjoyment therein.

## FRACTURE MECHANICS ON THE GO

In case you haven't noticed quite a bit has changed in the way scientific and technical research is being carried out compared to 20 years ago. In the early 1990's in order to research a technical topic one had to visit the nearest technical library and search through paper bound journals and hard bound books. The personal computer was very much in use at that time but employed mainly as a very advanced typewriter for desk top publishing or as a tool for computing and data analysis using spread sheet programs. However, the basic scientific and technical information required for engineering calculations and advanced analyses still had to be garnered from the books and journals sequestered in libraries.

Fast forward some 20 years and much of this has changed quite dramatically. Nearly everything one would search for in a library is now available on the internet searchable with a personal computer, a tablet style reader or even a cell phone. One has to wonder whether most students even know where the library is much less use it on a day to day basis.

In order to take advantage of all the newly available technology the MST web site will be

offering a new feature which will allow students and readers of the newsletter to perform some fairly sophisticated and useful fracture mechanics calculations related to the stability of coatings on surfaces and adhesion testing. In 2006 I published a hard bound volume on ADHESION MEASUREMENT METHODS: THEORY AND PRACTICE (CRC Press, 2006) which contained an entire chapter on the fracture mechanics of decohesion and delamination processes. In an effort to make this material more available to all those who have a need to determine whether the coatings they are dealing with will be stable or not this material will be made available on the MST web site in a format readily accessible not only to users of lap top computers but also to tablets and smartphone style equipment as well. As an example, figure (1) demonstrates a simple calculation to determine whether plasticity effects will be important in doing peel testing on metal coatings. Work by Farris and Goldfarb<sup>1</sup> demonstrated that when peeling common metals off rigid substrates up to 90% or more of the work done by the test machine goes into irreversible plastic flow and heat as opposed to overcoming the true metal/substrate interfacial energy. However, as shown on page 216 of the ADHESION MEASUREMENT METHODS volume there is a critical thickness above which plasticity effects no longer operate and the peel test will give the true metal/substrate interfacial energy. This thickness is given by the following formula:

$$t_c = \frac{3nEP}{\sigma_y^2} \quad (1)$$

Where:

- t<sub>c</sub> = Critical thickness
- E = Metal modulus
- P = Applied peel force per peel strip width
- n = Numerical factor in range 1-2

Figure (1) gives an example of how Eq.(1) can be rapidly evaluated to determine the critical thickness above which plasticity effects can be ignored for the case of copper coatings. Thus if a typical user is doing peel test measurements on copper films and is getting peel load values on order of 100 N/m and would like to know whether plasticity behavior is affecting his results he can bring up the web page on his smartphone and using the convenient slider

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<sup>1</sup> "An Experimental Partitioning of the Mechanical Energy Expended During Peel Testing", R. J. Farris and J. Goldfarb, in *Adhesion Measurement of films and Coatings*, K. L. Mittal, Ed. (VSP, Utrecht, The Netherlands, (1995), p265.

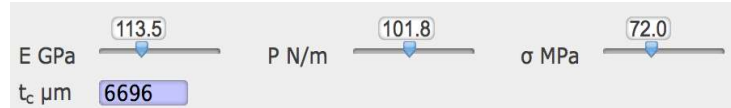
bar inputs evaluate Eq.(1) in a matter of seconds. The result for the critical thickness is given in the highlighted box and quickly shows that unless our user is dealing with very thick coatings his data will be affected by plasticity effects and his peel values will not reflect the true interfacial adhesion between the copper and the substrate.

This is a fairly simple example of formula evaluation using clever and efficient data input methods which make the analysis readily doable even on devices with very limited data input capabilities such as modern day smartphones.

Figure (2) gives an example of a much more sophisticated analysis which can be used to calculate whether or not a given coating will cause substrate decohesion. It is known, for example, that resins such as the epoxies when applied to brittle substrates such as glass can cause sever substrate spalling due to stress buildup in the epoxy caused by solvent loss shrinkage. Suo and Hutchinson ("Steady State Cracking in Brittle substrates Beneath Adherent films", Z. Suo and J. W. Hutchinson, *Int. J. Solids Structures*, 23, 1337 (1989)) have analyzed this problem and their results are discussed in detail in chapter 4 of the ADHESION MEASUREMENT METHODS volume.

Figure (2) shows the web page layout for a typical substrate decohesion calculation for the case of a polymer coating on a glass substrate. In this case a relatively thick coating of 175 microns is calculated to give a very dangerous driving force of  $2.33 \text{ MPa}\cdot\text{m}^{1/2}$  which will cause the substrate to crack as indicated by the red highlighted output box. Again this fairly complex calculation can be conveniently performed on tablet and smartPhone type equipment since all input is via slider boxes.

Before leaving this topic I would like to point out that this work has been done in cooperation with Prof. Steven Abbott who read the ADHESION MEASUREMENT METHODS volume and then contacted me pointing out that whereas the material therein was quite interesting and useful it would find a much greater audience if put on the web rather than gather dust on library shelves that almost no one visits anymore. I rather agreed with Prof. Abbott's observation and the initial results of our ongoing collaboration can be seen in figures (1-2). Please note that this is very much an ongoing project and the web pages have yet to be released for general use. A number of further topics have yet to be introduced and several corrections and improvements remain to be done. This topic will also be discussed in more detail in the upcoming short course on the

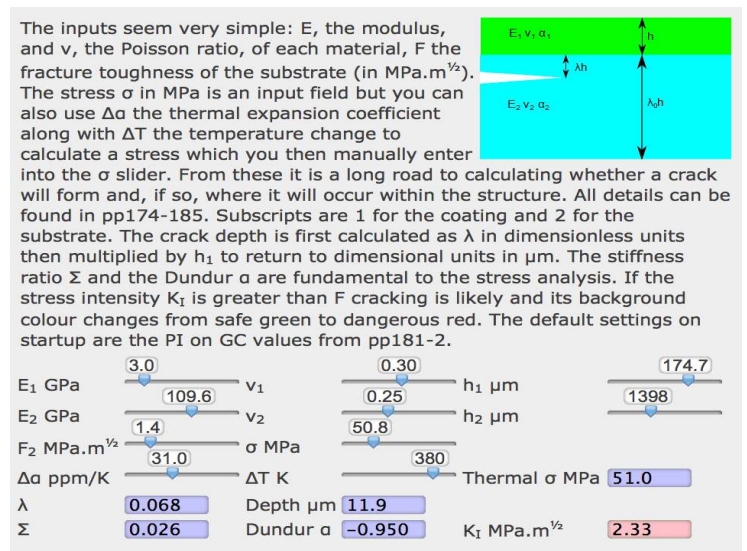


Note. Polymers can have rather more elastic recovery than metals (for which the formula is designed) so if you are thinner than  $t_c$  your  $W$  value may not be too far off. However, the point is that if you are above  $t_c$  you are safe, so the calculation is still insightful and the further you are below  $t_c$  the less accurate  $W$  will prove to be.

Here are a few typical values of  $E$  and  $\sigma$  for some standard materials:

Material	Modulus GPa	$\sigma$ MPa
Copper	110	70
Aluminum	70	150
Steel	200	250
Epoxy	2.5	55
PI	2.5	70
PET	4	95
PC	2.5	65
PMMA	2	60
PE	0.8	5

**Figure 1:** Sample of web page manipulation for evaluating Eq.(1) for the case of copper. Input is by handy slider bars. The modulus and yield stress are set close to values taken from the table and the peel load is taken at a nominal level close to 100N/m. The power law hardening exponent is set to 1 for this example. The critical thickness is given in the violet colored box. Note that the copper coating would have to be over 6.7mm thick to avoid plasticity effects upon peeling.



**Figure 2:** Sample web page to predict substrate decohesion due to coating stresses as formulated by the Suo-Hutchinson analysis. The case illustrated is for a 175 micron polymer coating on a glass substrate. The coating has an intrinsic stress level of 52 MPa due to thermal expansion mismatch with the glass. This stress level gives rise to a driving force of  $2.33 \text{ MPa}\cdot\text{m}^{1/2}$  which is predicted to give rise to substrate failure as indicated by the red highlighted output box.



CHEMISTRY, PHYSICS AND MECHANICS OF ADHESION SCIENCE To be held this coming October.

I should also point out that Prof. Abbott maintains his own web site covering a number of interesting and useful topics. It can be reached at:

<http://www.stevenabbott.co.uk/>

Topics on the web site include:

Coating and printing models  
Nano coatings and structures  
Hansen solubility Parameters  
Surface profiling  
Surfactants and solutions

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## ADHESION SCIENCE AND SILANE ADHESION PROMOTERS

We would like to announce two important events coming up this Fall in the scenic Hudson Valley New York. The first is the:

3- Day Impact Course

### [The Chemistry, Physics & Mechanics of Adhesion Science](#)

September 30- October 2, 2013, Hampton Inn, Harriman-Woodbury, New York

## Topics to be Covered

- I. Surface Contamination and Cleaning
- II. Theories or Mechanisms of Adhesion
- III. Contact Angle, Wettability and Adhesion
- IV. Investigation of Interfacial Interactions
- V. Surface Modification Techniques including Plasma
- VI. Ways to improve Adhesion of Organic Coatings
- VII. Silanes and Other Adhesion Promoters
- VIII. Adhesion Aspects of Thin Films
- IX. Adhesion Measurement of Films and Coatings
- X. Basics of Adhesion Measurement
- XI. Residual Stress and Material Mechanical Properties
- XII. Setting Adhesion Requirements for Coating Applications

- XIII. Adhesion Measurement at Atomic and Molecular Level
- XIV. Fundamental Adhesion Applications

## Adhesion's Important Role Today

Adhesion plays an important role in many technologies and industries, viz., aerospace, microelectronics, automotive, thin films, optics, coatings, paint and so on. Broadly speaking, the topic can be divided into two categories: film or coating/substrate combination, and adhesive joint. Films and coating are used for a variety of purposes and irrespective of their intended function, these must adhere adequately to the underlying substrate. So the need for understanding and controlling the factors affecting adhesion is quite patent.

Furthermore, the durability of the bond (on exposure to process chemicals, moisture, corrosives, etc.) is of grave concern and importance. This course presents an overview of the chemistry, physics and mechanics of adhesion in regard to understanding fundamental adhesion mechanisms. You will learn how to improve and control them and the latest adhesion measurement techniques which are being used to evaluate the PRACTICAL ADHESION of coatings and laminate structures.

Emphasis is given to methods which can be carried out in a manufacturing environment as well as in the lab environment; which give results that are directly relevant to the durability and performance of the structures under investigation. The effects of coating elastic properties and residual stress are considered as well as other external influences which affect durability under use conditions.

Audience: Scientists and professional staff in R&D, manufacturing, processing, quality control/reliability involved with adhesion aspects of coatings and adhesion sensitive applications.

Level: Beginner- Intermediate; introduction/overview

Prerequisites: Elementary background In chemistry, physics or materials science.

Duration: 3 days

Course fee and materials: \$1,295, includes break refreshments, complete set of lecture notes and copy of handbook and reference guide ADHESION MEASUREMENT METHODS: THEORY AND PRACTICE, (CRC PRESS, 2006)

## HOW YOU WILL BENEFIT FROM THIS COURSE

You will understand advantages and disadvantages of a range of adhesion measurement techniques. You will be able to select the right surface cleaning technique including the use of atmospheric plasma. You will utilize the concept of acid-base interactions in improving adhesion, and acquire basic skills for addressing adhesion failure problems. You will be able to analyze the alternatives and select the optimum technique for improving adhesion and durability. You will know where help is available in emergency situations and learn how to select best measurement technique for a given application.

## INSTRUCTORS AND CONTACT INFORMATION

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& Dr. R. H. Lacombe  
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E-mail: [klm@mstconf.com](mailto:klm@mstconf.com) ;  
[rhl@mstconf.com](mailto:rhl@mstconf.com)

For detailed information and registration:

[www.mstconf.com/AdhesionCourse.htm](http://www.mstconf.com/AdhesionCourse.htm)

## NINTH INTERNATIONAL SYMPOSIUM ON SILANES AND OTHER COUPLING AGENTS

To be held October 3-4, 2013, at the Hampton Inn, Harriman-Woodbury, New York

## SYMPOSIUM HISTORY AND MOTIVATION

This symposium continues the tradition set by the first symposium in this series: "Silanes and Other Coupling Agents" which was hosted in 1991 by the Dow Corning Corporation in honor of Dr. Edwin P. Plueddemann. As with its predecessors, this symposium will be concerned with the technological areas where the use of surface primers such as silanes are critical to the success of many technologies.

Historically the silanes have been used as coupling agents for thin films in the microelectronics industry and in glass fiber composites where the use of silanes has been an enabling factor in the success of many manufactured products. Quite surprisingly, silanes have also found a role in biotechnology as specific coupling agents for

bonding polynucleotides to the so-called "gene chips" and also in cosmetic applications.

## AUDIENCE AND PARTICIPATION

This symposium is organized to bring together scientists, technologists and engineers interested in all aspects of silanes and other coupling agents, to review and assess the current state of knowledge, to provide a forum for exchange and cross-fertilization of ideas, and to define problem areas which need intensified efforts.

## AMONG TOPICS TO BE COVERED ARE:

- ▶ Mechanisms of silane action.
- ▶ Role of silanes in adhesion of coatings, composites and adhesive joints.
- ▶ Deposition techniques:
  - solution
  - plasma
  - vapor
  - electrochemical
- ▶ Non-silane adhesion promoters.
- ▶ Plasma polymerized coatings as adhesion promoters.
- ▶ Relevance of silanes in durability of bonds.
- ▶ Applications:
  - coatings, corrosion inhibitors
  - adhesive joints, composites
  - biological applications: dental, gene chips, cosmetics.
- ▶ Silane surface characterization.
  - Standard: contact angle, FTIR,...
  - Advanced: neutron scattering, ...

## SUBMITTING A PAPER

This symposium is being organized by MST Conferences under the direction of Dr. K. L. Mittal, Editor, Reviews of Adhesion and Adhesives. Please notify the conference chairman of your intentions to present a paper as early as possible. An abstract of about 200 words should be sent by August 31, 2013 to the conference chairman by any of the following methods:

E-mail: [rhl@mstconf.com](mailto:rhl@mstconf.com)  
FAX: 212-656-1016

Regular mail:

Dr. Robert H. Lacombe  
Conference Chairman  
3 Hammer Drive  
Hopewell Junction, NY 12533

Contact by phone: 845-897-1654; 845-227-7026  
Full conference details and registration via the  
Internet will be maintained on our web site:

<http://mstconf.com/silanes9.htm>

CANCELLATIONS: Registration fees are refundable, subject to a 15% service charge, if cancellation is made by September 15, 2013. NO refunds will be given after that date. All cancellations must be in writing. Substitutions from the same organization may be made at any time without penalty. MST Conferences reserves the right to cancel the symposium or the short course if it deems this necessary and will, in such event, make a full refund of the registration fee. No liability is assumed by MST Conferences for changes in program content.

#### DATES:

September 30 - October 2, 2013

3- Day Impact Course

THE CHEMISTRY, PHYSICS & MECHANICS  
OF ADHESION SCIENCE

October 3-4, 2013

NINTH INTERNATIONAL SYMPOSIUM ON  
SILANES AND OTHER COUPLING AGENTS

#### LOCATION

Hampton Inn Harriman Woodbury  
60 Centre Drive  
Central Valley,  
New York, 10917, USA

TEL: 1-845-782-9600

Web site:

<http://hamptoninn3.hilton.com/en/hotels/new-york/hampton-inn-harriman-woodbury-NYCMRHX/index.html>

#### TO REGISTER

BY PHONE: 845-897-1654; 845-227-7026

BY FAX: 212-656-1016

E-mail: [rhl@mstconf.com](mailto:rhl@mstconf.com)

ONLINE:

[www.mstconf.com/RegMST.htm](http://www.mstconf.com/RegMST.htm)

BY MAIL: SEND COMPLETED FORM BELOW TO

Dr. Robert Lacombe  
Chairman  
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Hopewell Junction, NY 12533-6124, USA



REGISTRATION FORM:

SHORT COURSE ON CHEMISTRY, PHYSICS AND MECHANICS OF ADHESION SCIENCE, September 30 - October 2, 2013	\$1295
SYMPOSIUM ON SILANES AND OTHER OTHER COUPLING AGENTS, October 3-4, 2013, Student-Presenter	\$395
SYMPOSIUM ON SILANES AND OTHER OTHER COUPLING AGENTS, October 3-4, 2013, Regular attendee	\$695
Deduct 10% if more than 1 participant from same institution	
TOTAL REGISTRATION FEE	

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CREDIT CARD: Check here and fill out box below	
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CHECK: Make check payable to MST Conferences, LLC and mail to: Dr. Robert H. Lacombe Chairman 3 Hammer Drive Hopewell Junction, NY 12533-6124, USA	

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2-For all residing in the continental US, when paying by credit card the address should be the card billing address