DOE/SF/19460--299-Pt.5

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1998 SUMMER RESEARCH PROGRAM FOR HIGH SCHOOL JUNIORS

AT THE

UNIVERSITY OF ROCHESTER'S

LABORATORY FOR LASER ENERGETICS

STUDENT RESEARCH REPORTS

PROJECT COORDINATOR

Dr. R. Stephen Craxton

March 1999

Laboratory Report 300

Laboratory for Laser Energetics
University of Rochester

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During the summer of 1998, 11 students from Rochester-area high schools participated in the Laboratory for Laser Energetics' Summer High School Research Program. The goal of this program is to excite a group of high school students about careers in the areas of science and technology by exposing them to research in a state-of-the-art environment. Too often, students are exposed to "research" only through classroom laboratories that have prescribed procedures and predictable results. In LLE's summer program, the students experience all of the trials, tribulations, and rewards of scientific research. By participating in research in a real environment, the students often become more excited about careers in science and technology. In addition, LLE gains from the contributions of the many highly talented students who are attracted to the program.

The students spent most of their time working on their individual research projects with members of LLE's technical staff. The projects were related to current research activities at LLE and covered a broad range of areas of interest including optics, spectroscopy, chemistry, diagnostic development, and materials science. The students, their high schools, their LLE supervisors and their project titles are listed in the table. Their written reports are collected in this volume.

The students attended weekly seminars on technical topics associated with LLE's research. Topics this year included lasers, fusion, holography, nonlinear optics, global warming, and scientific ethics. The students also received safety training, learned how to give scientific presentations, and were introduced to LLE's resources, especially the computational facilities.

The program culminated with the High School Student Summer Research Symposium on 26 August at which the students presented the results of their research to an audience that included parents, teachers, and members of LLE. Each student spoke for approximately ten minutes and answered questions. At the symposium an Inspirational Science Teacher award was presented to Mr. David Crane, a chemistry teacher at Greece Arcadia High School. This annual award honors a teacher, nominated by alumni of the LLE program, who has inspired outstanding students in the areas of science, mathematics, and technology.

High School Students and Their Projects (1998)			
Student	**High School**	**Supervisor**	**Project**
Steven Corsello	Pittsford Mendon	K. Marshall	Computer-Aided Design and Modeling of Nickel Dithiolene Near-Infrared Dyes
Peter Grossman	Wilson Magnet	R. S. Craxton	Group Velocity Effects in Broadband Frequency Conversion on OMEGA
Joshua Hubregsen	Pittsford Sutherland	S. Jacobs	A Study of Material Removal During Magnetorheological Finishing (MRF)
Nieraj Jain	Pittsford Sutherland	M. Guardelben	Analyzing Algorithms for Nonlinear and Spatially Nonuniform Phase Shifts in the Liquid Crystal Point Diffraction Interferometer
Leslie Lai	Pittsford Mendon	M. Wittman	The Use of Design-of-Experiments Methodology to Optimize Polymer Capsule Fabrication
Irene Lippa	Byron-Bergen	K. Marshall	Synthesis and Analysis of Nickel Dithiolene Dyes in a Nematic Liquid Crystal Host
Phillip Ostromogolsky	Brighton	F. Marshall	Investigation of the X-Ray Diffraction Properties of a Synthetic Multilayer
Michael Schubmehl	The Harley School	R. Epstein	An Analysis of the Uncertainty in Temperature and Density Estimates from Fitting Model Spectra to Data
Joshua Silbermann	Penfield	P. Jannimagi	Automated CCD Camera Characterization
Abigail Stern	The Harley School	J. Knauer	Design and Testing of a Compact X-Ray Diode
Amy Turner	Churchville-Chili	R. S. Craxton	Ray Tracing Through the Liquid Crystal Point Diffraction Interferometer

A total of 91 high school students have participated in the program since it began in 1989. The students this year were selected from approximately 60 applicants. Each applicant submitted an essay describing their interests in science, a copy of their transcript, and a letter of recommendation from a science or math teacher.

LLE plans to continue this program in future years. The program is strictly for students from Rochester-area high schools who have just completed their junior year. Applications are generally mailed out in February with an application deadline near the end of March. For more information about the program or an application form, please contact Dr. R. Stephen Craxton at LLE.

This program was supported by the U.S. Department of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-FC03-92SF19460.

THE USE OF DESIGN-OF- EXPERIMENTS METHODOLOGY TO OPTIMIZE POLYMER CAPSULE FABRICATION

by

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The Use of Design-of-Experiments Methodology to Optimize Polymer Capsule Fabrication

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Laboratory for Laser Energetics
University of Rochester

Summer High School Research Program
1998

Abstract

Future inertial-fusion experiments on Omega will utilize ~1-mm-diameter cryogenic targets that have a ~100-μm-thick, uniformly-frozen fuel layer on their interior. It is desired that they have a stress-free wall thickness <1 μm and an rms surface roughness <20 nm. A design-of-experiments (DOE) approach was used to characterize a glow-discharge-polymerization coater built at LLE to fabricate smooth, stress-free capsules with submicron wall thicknesses. The DOE approach was selected because several parameters can be changed simultaneously in a manner which allows the minimum number of runs to be performed to obtain statistically-relevant data. Planar, silicon substrates were coated with ~3-5 μm of polymer and profilometry was used to determine the coating rate, the film stress, and the surface roughness. The coating rate was found to depend on the trans-2-butene/hydrogen ratio, the total gas-flow rate, the total chamber pressure, and the RF power. In addition, a two-parameter interaction between the total pressure and the RF power also affects the coating rate. The film stress depends on the total chamber pressure and the total mass-flow rate. The surface roughness is independent of the parameters studied. Preliminary results indicate that capsules can be produced rapidly without affecting the smoothness of their outside surface and without residual stress in their walls.

Introduction

The main charter of the Laboratory for Laser Energetics (LLE) at the University of Rochester is to investigate inertial-confinement fusion (ICF) using the direct-drive approach. This is achieved by uniformly illuminating a target with the sixty beams of the 30 kJ Omega laser thereby compressing its contents to a density and pressure sufficient to initiate nuclear fusion.

Targets typically consist of a spherical polymer capsule that is ~1 mm in diameter with a wall thickness of ~10-30 μm. The capsule is diffusion filled with the hydrogen isotopes deuterium and tritium, and is then coated with a 1000-A-thick aluminum layer to retain the gas in the capsule and prevent preheat of the capsule during the early stages of the laser pulse. Future experiments will utilize cryogenic targets that have a ~100-μm-thick, uniformly-frozen fuel layer on their interior. It has been determined theoretically that these targets will perform optimally if their wall thickness is less than 1 μm and their rms surface roughness is <20 nm.

The ultimate goal of the coating experiments described here is to fabricate stress-free capsules with submicron wall thicknesses. The capsules used in current Omega experiments are fabricated using a glow-discharge-polymerization (GDP) process.[1] A GDP coater was built at LLE specifically for this purpose. However, it differs considerably from designs currently being used by other target-fabrication labs who continue to use coaters based on technology developed in the 1980s. Therefore, to first understand the GDP coating process in our coating geometry, a series of runs were performed in which polymer films were deposited onto planar silicon substrates. To this end, the widely-accepted, statistically-rigorous design-of-experiments (DOE) method[2,3] was used to identify parameters affecting the GDP process by measuring the properties of the films produced.

Construction of the GDP coater

The design and construction of the GDP coater is shown in Figure 1. Trans-2-butene ($HCH_3C{=}CH_3CH$) is the hydrocarbon gas used as the precursor for the coating material. This is introduced into a hydrogen carrier-gas stream to maintain a saturation of hydrogen ions in the resultant plasma. This assures that the deposited GDP films with have nearly a 1:1 carbon to hydrogen ratio. The individual gas-flow rates and the concentration ratio between the gases are regulated by flow controllers (Unit, Model 8100 with Model URS-100-5 readout). The gas mixture enters a quartz tube which is surrounded by a helical-resonator.[4,5] The helical-resonator is driven at its resonant frequency (11.77 MHz) by amplifying (ENI, Model A150) the output of a function generator (Hewlett-Packard, Model 33120A). The high-voltage, radio-frequency electric field causes the gases in the quartz tube to ionize into chemically-active species. Primarily, the double bond in the trans-2-butene dissociates into HCH_3C^+, which then deposits onto the substrate. Unsatisfied chemical bonds on the substrate either bond with these ions or hydrogen as the material deposits. The process is enclosed in a vacuum chamber which is pumped by a high-throughput mechanical pump with a roots blower (Edwards, E2M40 and EH250). The pressure in the chamber is monitored by a capacitance manometer (MKS, Type 621) which communicates with a downstream pressure-control valve (MKS, Type 653 with 600 Series controller) to actively control the chamber's pressure.

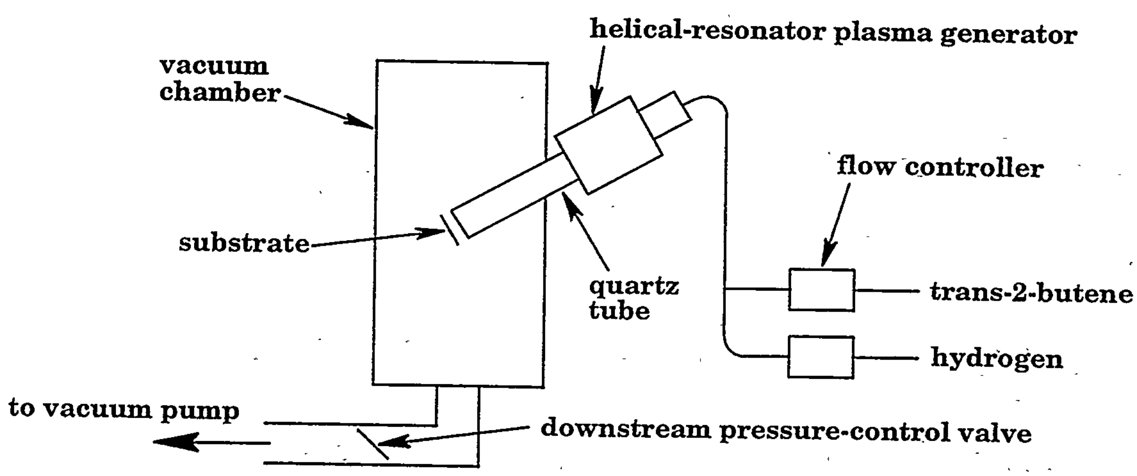

Figure 1. A schematic of the coating chamber and its components. The coater is operated using active-feedback controls which assures stability of the coating parameters during the deposition process.

Method

Historically, experiments have been performed using "trial-and-error" or "one-parameter-at-a-time" techniques. However, these techniques have several weaknesses. Trial-and-error methods proceed by randomly sampling the parameter space, produce unevenly distributed data, and seldomly generate statistically-meaningful conclusions. Also, regions of the parameter space may be entirely neglected; this makes process optimization uncertain. Changing one parameter at a time while leaving the others fixed requires numerous experiments and does not allow examination of significant interactions between the individual parameters. Also, if there is an important but uncontrolled parameter in an experiment, such as ambient humidity or operator variability, one-parameter-at-a-time experiments are susceptible to error or can be inconclusive altogether.

The DOE approach was selected for this experiment because of its particular advantages. Most importantly, several parameters can be changed simultaneously in a manner which allows *the minimum number of runs to be performed to obtain statistically-relevant data.* Initial screening experiments are first designed and performed to determine the statistically-significant parameters that affect the process under investigation. Quantifiable properties of the product termed "responses" are measured after each experimental run. After the runs are completed, "analysis-of-variance" (ANOVA) techniques are applied to determine statistically-significant parameters. Conclusions from this data can be drawn at a quantifiable confidence level (usually >95%) with a minimum number of runs because of DOE's statistically-rigorous methodology. Once the important process parameters are determined, the initial set of experiments is then augmented by performing additional experiments outside boundaries of the original parameter space. These results are then used to produce "response surfaces" for process optimization.

PARAMETER	UNITS	LOW VALUE	CENTER VALUE	HIGH VALUE
Total chamber pressure	mTorr	32.50	55.0	77.50
Trans-2-butene / H_2 ratio	%	3.25	5.50	7.75
Total gas-flow rate	std cc/min	3.75	5.00	6.25
RF plasma power	Watts	20.0	30.0	40.0

Table 1. The parameters studied and their values used in the factorial set of experiments, including their centerpoint values.

For this particular investigation, the effect of four externally-controllable parameters on coating rate, film stress, and surface roughness were studied, as shown in Table 1. The experiments were designed and analyzed using a commercially-available software package.[6] A set of 16 "two-level factorial" experiments were designed which included all possible combinations of extreme values of the parameters studied (see Appendix). In addition, four "center point" runs were included with all of the parameters set at the midpoint between their extremes. The order of these 20 runs were then randomized. By systematically repeating the center-point run at random intervals throughout the set of runs, the reproducibility of the process during the course of the runs is quantified into a "pure error" that can be attributed to the process. This differs from determining the measurement error for each response (by repeatedly remeasuring an individual sample) since the entire process is repeated. A subset of these runs is depicted graphically in Figure 2.

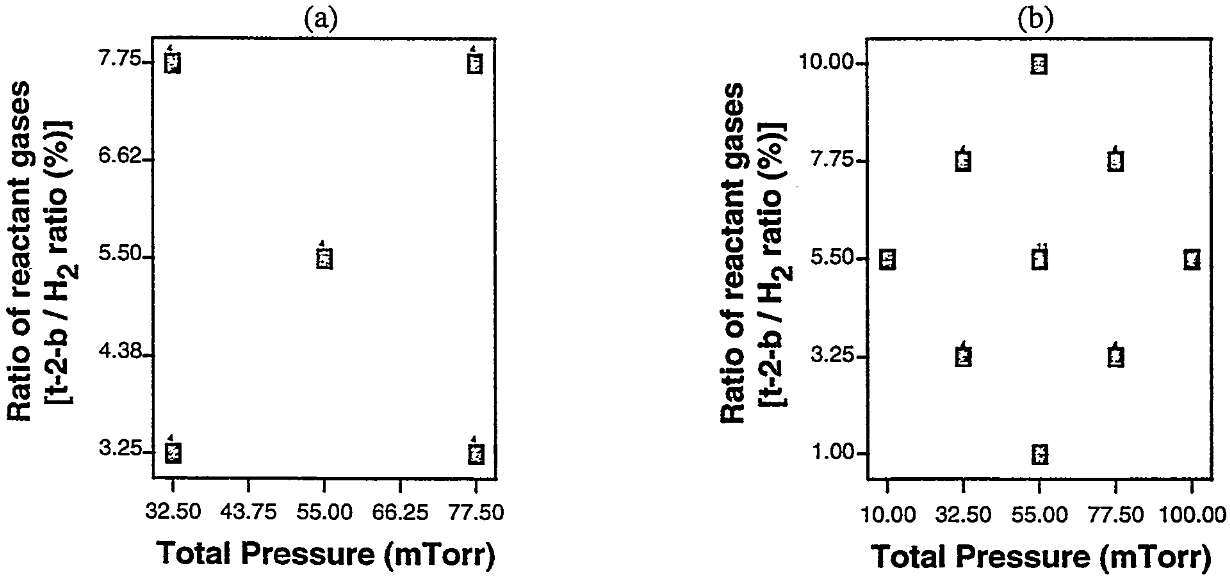

Figure 2. (a) The segment of the fractional factorial design that included varying the total pressure and the ratio of the reactant gases. The center point is also depicted. The small numbers above each point designate the number of runs that were performed that included these values for the displayed parameters, i.e., the runs including the extreme values for the other two parameters have been projected onto the plane shown. (b) The CCD design obtained by augmenting the design shown in (a).

Two clean silicon substrates were coated during each run: one ~4x25 mm that was masked along a 3 mm section at its center and at one end, and a narrow (>10:1 aspect ratio) beam with a length ~25 mm. The surface profile of the narrow beam was traced by the Rank Taylor Hobson Form Talysurf Series profilometer over its length ℓ before the coating run to determine its initial curvature. The duration of each experimental run was 6-8 hr to produce a coating ~3-5 μm thick.

The coating rate was determined by removing the masks from the large silicon substrate and measuring the step height of the film with the profilometer. This film thickness t_{film} was divided by the coating time to determine the coating rate. The surface roughness of the film was then measured with a Zygo NewView scanning white-light interferometer. This yielded a 2-dimensional profile of the film's surface and allowed the rms roughness to be determined along lines in multiple directions, which were then averaged.

To determine the film stress, the thickness $t_{substrate}$ of the narrow silicon beam was measured with a micrometer capable of 1 μm resolution. After coating, the narrow beam was again traced over a length ℓ with the profilometer to find its final curvature. The difference between the initial and final profile heights **Δh** over the length ℓ was used to determine the change in the substrate's radius of curvature **ΔR** due to forces imposed by the film on its surface. The film stress σ_{total} is found from:[7]

$$\sigma_{total} = [E_s / 6(1-\nu_{\sigma})][t^2_{substrate} / t_{film}][1 / \Delta R]$$

where E_s and ν_s are the elastic modulus and Poisson ratio for the substrate respectively ($E_s / (1 - \nu_s) = 1.805\text{x}10^5$ MPa for silicon along the <100> crystal direction),

and $\Delta R = \ell^2 / 8(\Delta h)$. A negative value for film stress implies compressive stress in the film whereas a positive value implies tensile stress.

Results

Data gathered from the experimental runs were analyzed using analysis of variance (ANOVA) techniques. Figure 3(a) shows the effect of the ratio between trans-2-butene and hydrogen on the coating rate and Figure 3(b) shows the effect of the total-flow rate on the coating rate. In Figure 3(a), the lower I-bar signifies the mean of all the coating rates from runs with a low trans-2-butene/hydrogen ratio. The higher I-bar represents the mean of all coating rates from runs with a high trans-2-butene/hydrogen ratio. The I-bars in Figure 3(b) represent the effect of the flow rate in a similar fashion. The I-bars in each plot represent the fluctuation of the response data gathered at each extreme value within a 95% confidence level. Given the vertical displacement of the I-bars at each extreme, there is >99.99% confidence that both parameters have significant effects on the GDP coating rate.

If the difference between the average coating rates at the high and low values of each parameter is plotted as a normal distribution, an "effects plot" is generated as shown in Figure 4. The parameters that lie on the line can be explained by random fluctuation in the data, whereas parameters that deviate from the line have a statistically significant effect on the process. The triangular points are derived from the repeated "center points" and their deviation from the line represents the experimental error of the data; insignificant parameters and their interactions lie as close to the line as these triangles do. As shown before, the trans-2-butene/hydrogen ratio and the total-flow rate both have a significant effect on the process, as do the total pressure and the RF power. Figure 4 also indicates the significance of a two-parameter interaction between the total pressure and the RF power, a two-parameter interaction between the trans-2-butene/hydrogen ratio and the total-flow rate, and a three-parameter interaction.

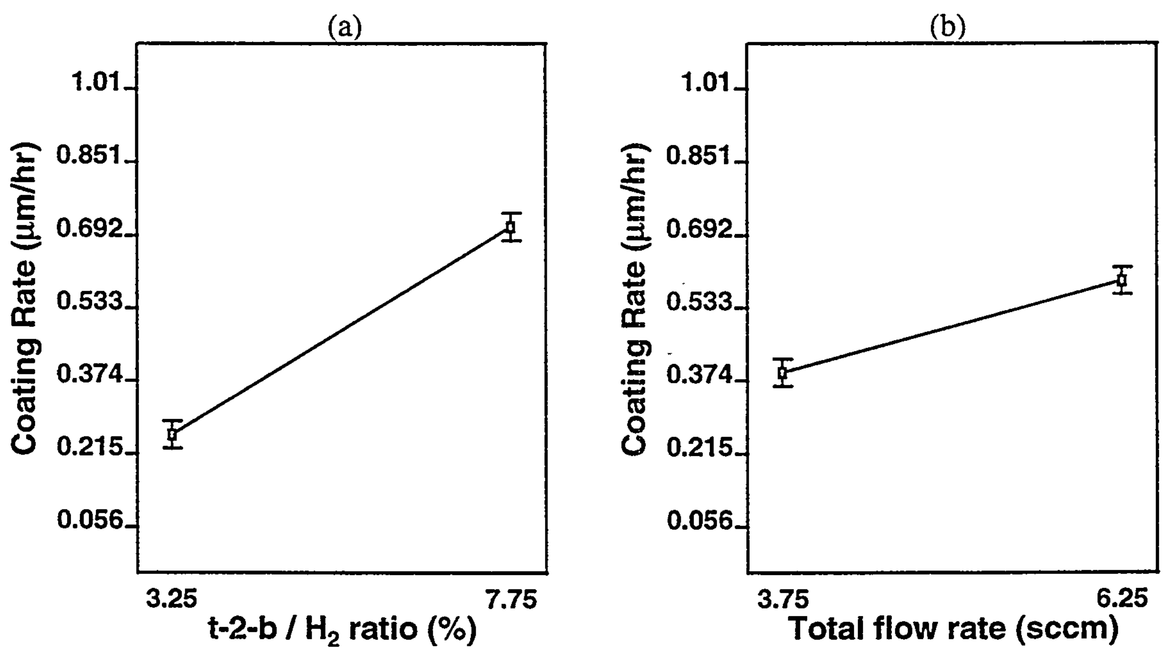

Figure 3. (a) The effect of the ratio between trans-2-butene and hydrogen on the coating rate and (b) the effect of the total-flow rate on the coating rate.

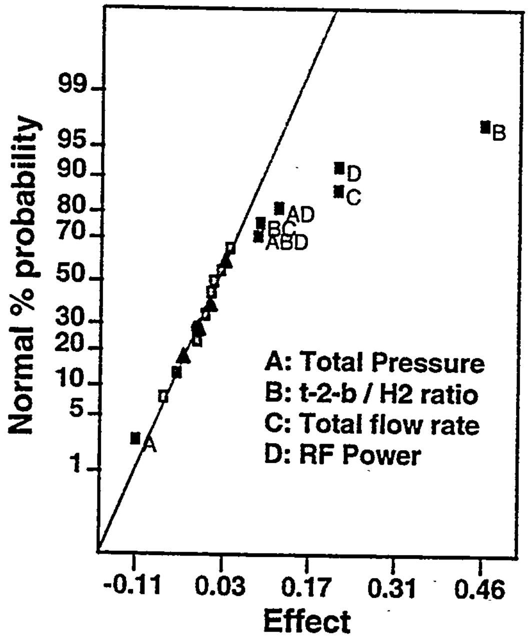

Figure 4. Normally-distributed effects plot of the parameters affecting the coating rate. The parameters/interactions that have a significant (>95% confidence interval) effect on the process deviate from the line that best fits the remaining, insignificant parameters/interactions. Note that the total chamber pressure has a negative effect, i.e., an increase in pressure results in a decreased coating rate. Three-factor interactions are rare; indeed the ABD interaction shown was proved to be insignificant when the study was augmented to a CCD design.

The effects plots of film stress and surface roughness shown in Figure 5 indicates that they are not significant functions of the parameters investigated. The deviation of the parameters/interactions from the line is roughly equal to the deviation of the triangular repeated "center points" and can thus be explained by random fluctuations in the data. Since no parameter deviates considerably from either line, it can be concluded that no parameter has a statistically-significant effect on the film stress or the surface roughness. However, from these factorial-design experiments, the average film stress was found to be -101^{+}/-30 MPa and the average surface roughness was found to be 4.5^{+}/-1.0 nm, rms.

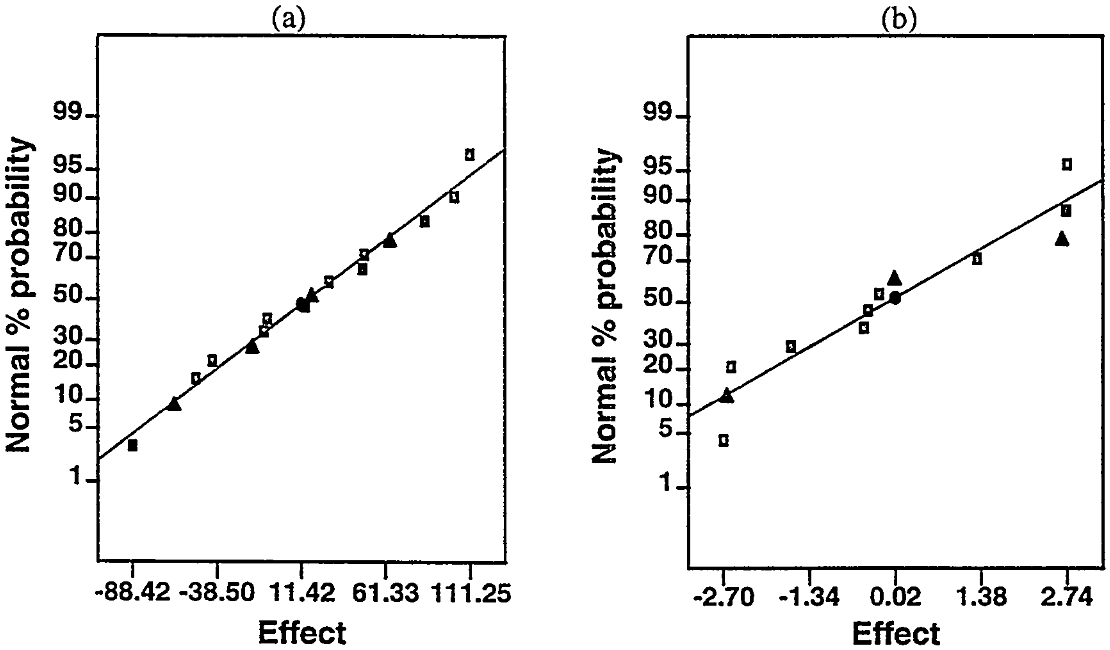

Figure 5. The effects plots of (a) film stress and (b) surface roughness. No parameters/interactions are statistically significant.

Once the significant parameters were determined for the responses being studied, the entire factorial design was augmented to a central-composite design (CCD) to obtain a mathematical model of the process (see Appendix). Figure 2(b) shows the additional "star" or "axial" points that were added to the original set of experiments: 8 plus two center-point repeats. The star points in the CCD allow the parameter space outside the original zone to be examined, thus reducing the uncertainty of the model near the edges of parameter space. In addition, quadratic surfaces can be fit to the data since multiple lines can be drawn in Figure 2(b) that intersect three sets of runs. Local minima and maxima can then be found on the second-order polynomial surfaces allowing optimization of the process.

A quadratic model was found to best fit the coating-rate data and is given by: Coating Rate = -0.21 - (0.011 * Total Pressure) + (0.040 * t-2-b / H_2 ratio) + (0.012 * Total-flow rate) + (0.017 * RF Power) - (3.655E-04 * RF Power^2)+ (2.970E-04 * Total Pressure * RF Power) +

(0.014 * t-2-b / H2 ratio * Total-flow rate). The units for coating rate is μm/hr and units for the individual parameters are given in Table 1. The power-pressure two-parameter interaction revealed in Figure 4 is evident in the coating-rate response surface shown in Figure 6. An increase in total pressure at a high RF power (40 watts) increases the coating rate, whereas an increase in total pressure at a low RF power (20 watts) decreases the coating rate.

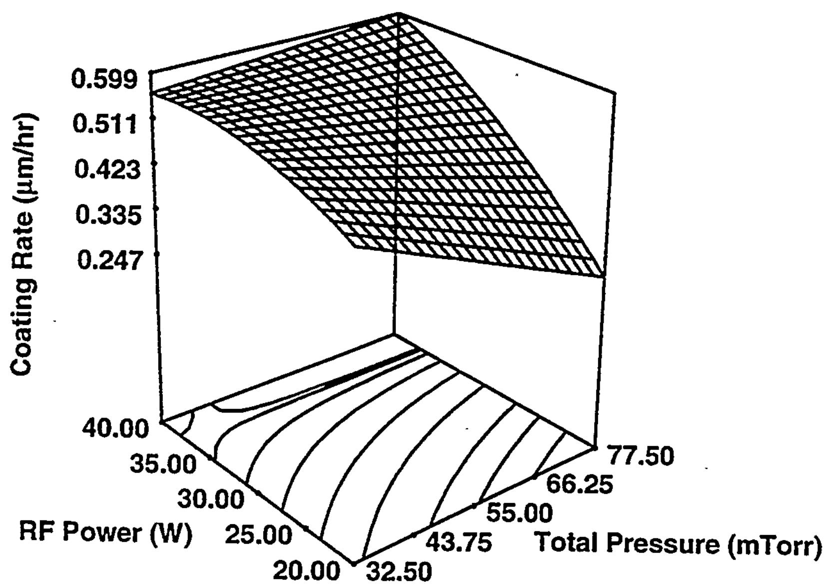

Figure 6. The coating-rate response surface as a function of RF power and total chamber pressure. The trans-2-butene / H_2 ratio = 5.50 % and the total-flow rate = 5.00 sccm for this plot.

The additional data collected for the CCD revealed a dependence of film stress on the total pressure and the total mass-flow rate as shown in Figure 7. A linear model best fit the film-stress data given by: Film Stress = -405.66 + (1.80 * Total Pressure) + (35.88 * Total-flow rate) where the units for film stress are MPa. The additional surface-roughness data confirmed that the roughness of the film is independent of the parameters studied, with an average of 3.5+/-0.6 nm, rms. The average surface roughness from the CCD design agrees with the average from the factorial design within their respective standard deviations.

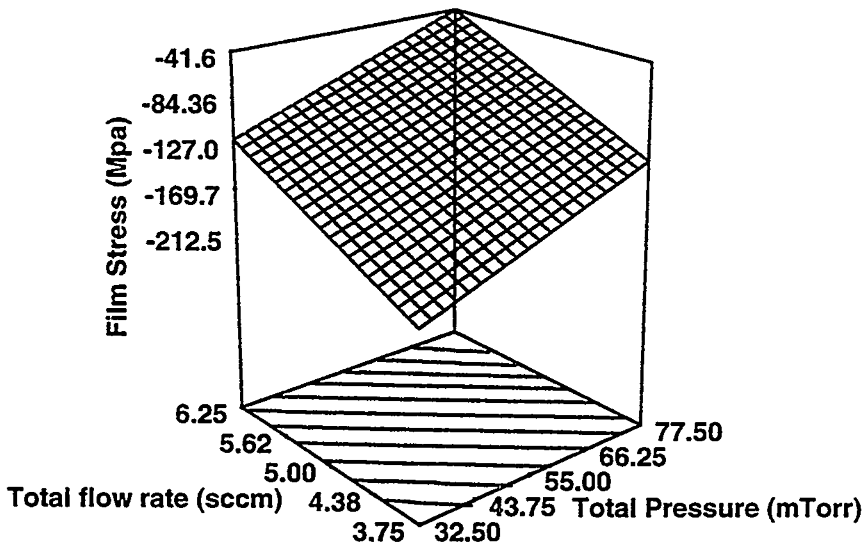

Figure 7. The film-stress response surface as a function of trans-2-butene/hydrogen ratio and the total mass-flow rate.

Conclusion

Although the effects of these parameters on the properties of the deposited films were determined empirically, the results of this study make sense scientifically. The coating rate should be proportional to the trans-2-butene/hydrogen ratio, the total mass-flow rate, and the RF power delivered to the plasma, since to first two parameters control how much raw material is delivered to the substrate and the latter controls the excitation rate of the chemically-active species. The two-factor interaction shown in Figure 6 between the total pressure in the chamber and the RF power can be explained as follows. The collision frequency of the molecules in a gas is proportional to its pressure; in the plasma, the collision frequency is proportional to the de-excitation and reaction rates. An increase in pressure increases the de-excitation rate of the molecules, but at low power, the excitation rate is low. The active species collide with the wall of the quartz tube or react with one another in the gas phase before reaching the substrate, thereby reducing the coating rate. At higher power, the excitation rate is sufficient to activate the additional molecules that are introduced as the pressure increases. The high excitation rate compensates for the increased collision frequency, thereby increasing the coating rate. Note that as the pressure is increased, the increase in coating rate at high power is not as dramatic as its reduction at low power.

The film stress and coating rate are both proportional to the total-flow rate and the total pressure (at higher RF powers). It is conceivable the film stress can be reduced to zero by exploring the parameter space in regions of higher total-flow rates and total pressures. Additionally, surface roughness is independent of the parameters investigated. Therefore, preliminary results indicate that capsules can be produced rapidly without affecting the smoothness of their outside surface and without residual stress in their walls.

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Acknowledgments

I would like to thank Dr. R. S. Craxton for organizing this unique program and for giving me the opportunity to be part of it. Special thanks goes to the Micro-fabrication Laboratory which allowed me extensive use of their facility. Finally, I would like to express my deep appreciation to my advisor Mark Wittman, who spent countless hours designing this project, building the GDP coater, teaching me how to use all the lab equipment, and making this experience memorable.

Appendix

The table below is the raw data collected during these experiments. The asterisk (*) indicates that data was not obtained for that run for one of two reasons: the film peeled from the substrate due to excessive film stress or inadequate substrate preparation, or the film was too thin to measure its surface roughness with the white-light interferometer due to interference from reflections from the substrate. The double asterisk (**) indicates that this data was removed from the analysis since the software indicated it was an "outlier" from the rest of the data, i.e., it was more than 3.5 standard deviations from the ensemble mean of the response. The dark line delineates the original factorial design from the points added to complete the CCD. Run # 18 was accidentally duplicated during the factorial set of experiments instead of performing run # 19. The original run # 19 was added to the end of the experiment as run # 31.

RUN #	POINT TYPE	TOTAL CHAMBER PRESSURE (mTorr)	TRANS-2-BUTENE / H_2 RATIO (%)	TOTAL GAS-FLOW RATE (std cc/min)	RF PLASMA POWER (Watts)	COATING RATE (µm/hr)	FILM STRESS (MPa)	SURFACE ROUGHNESS (nm)
1	Factorial	77.5	7.75	3.75	20.0	0.306	-177	*
2	Factorial	77.5	7.75	3.75	40.0	0.655	-42.4	3.98
3	Factorial	77.5	3.25	6.25	40.0	0.407	36.8	1.68
4	Center	55.0	5.50	5.00	30.0	0.500	-81.2	2.21
5	Factorial	32.5	3.25	6.25	40.0	0.402	-109	7.42
6	Factorial	77.5	7.75	6.25	40.0	1.01	18.7	4.32
7	Center	55.0	5.50	5.00	30.0	0.479	-337	11.1
8	Factorial	32.5	7.75	6.25	20.0	0.957	-98.9	*
9	Factorial	32.5	3.25	6.25	20.0	0.279	638**	1.42
10	Factorial	32.5	3.25	3.75	20.0	0.056	*	*
11	Center	55.0	5.50	5.00	30.0	0.457	-219	2.93
12	Factorial	32.5	3.25	3.75	40.0	0.273	-95.0	7.56
13	Factorial	32.5	7.75	3.75	40.0	0.600	*	*
14	Factorial	77.5	3.25	3.75	40.0	0.254	-198	9.40
RUN #	POINT TYPE	TOTAL CHAMBER	TRANS-2-BUTENE /	TOTAL GAS-FLOW RATE	RF PLASMA	COATING RATE	FILM STRESS	SURFACE ROUGHNES

		PRESSURE (mTorr)	H_2 RATIO (%)	(std cc/min)	POWER (Watts)	(µm/hr)	(MPa)	S (nm)
15	Center	55.0	5.50	5.00	30.0	0.400	-290	*
16	Factorial	32.5	7.75	6.25	40.0	0.971	-116	2.04
17	Factorial	32.5	7.75	3.75	20.0	0.600	-359	*
18	Factorial	77.5	7.75	6.25	20.0	0.594	-71.8	3.79
19	Factorial	77.5	7.75	6.25	20.0	0.462	-119	3.73
20	Factorial	77.5	3.25	6.25	20.0	0.122	17.6	0.474
21	Axial	100.0	5.50	5.00	30.0	0.365	-66.2	1.73
22	Axial	55.0	5.50	5.00	50.0	0.571	-83.9	1.21
23	Axial	10.0	5.50	5.00	30.0	0.479	-136	1.79
24	Axial	55.0	5.50	2.50	30.0	0.296	*	4.48
25	Axial	55.0	5.50	5.00	10.0	0.144	-114	3.46
26	Axial	55.0	5.50	7.50	30.0	0.716	-104	3.55
27	Axial	55.0	10.00	5.00	30.0	1.15	-115	*
28	Axial	55.0	1.00	5.00	30.0	0.0486	-160	0.652
29	Center	55.0	5.50	5.00	30.0	0.569	-91.1	*
30	Center	55.0	5.50	5.00	30.0	0.558	-79.7	2.92
31	Factorial	77.5	3.25	3.75	20.0	0.0861	-37.7	*