MANUFACTURING AND PERFORMANCE EVALUATION OF CARBON NANOTUBE-PARYLENE SANDWICH THIN FILMS

by

Chia-Ling Chen, Ernesto Lopez, Yung-Joon Jung,
Mehmet R. Dokmeci and Sinan Müftü
Northeastern University
USA

ABSTRACT

Parylene-C, an inert and relatively mechanically strong polymer, which can be deposited in a conformal manner, is a promising substrate candidate for flexible electronics (Flextronics) devices. Parylene-CNT sandwich-films were fabricated by utilizing single-walled carbon nanotube (SWNT) layers sandwiched between two, 10 µm thick parylene layers. The device was fabricated using shadow mask technology and SWNT drop casting on top of the Parylene substrate. The electrical conductivity and mechanical properties of the samples were tested under tensile loading. The load-unload tests showed small change in electrical resistance (~1%) when applying a strain in the range 0 - 2%, with negligible hysterisis. The tensile test also showed ~32% increase in the elastic modulus (E) of the sandwich film, relative to pure parylene. Potential applications are in interconnects for flexible electronics devices, and strain sensors for biological systems.

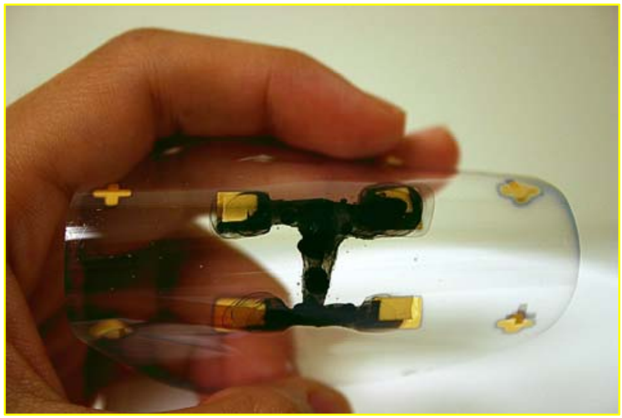
INTRODUCTION

There is a growing interest in making sensors, optoelectronic and electronic devices on flexible polymer films [1-3]. Interconnects with lower resistance are desirable since they result in lower power consumption and increased device speeds. Carbon nanotubes (CNTs), first discovered by Iijima in 1991 [4], are excellent candidates for interconnects due to their low electrical and thermal resistance, and stability at high temperature and current [5]. With their exceptionally high mobility exceeding 75,000cm^2/Volt-sec, CNTs can also be utilized in active device applications [6]. Furthermore, the hollow structure and closed topology allow extreme strains under tension (40%) without showing signs of brittle behavior, plastic deformation, or bond rupture [7-8]. Besides, metal interconnects scaled in size fail rather easily due to electromigration phenomena, which is eliminated in covalently bonded CNTs because there are no low-energy defects or dislocations that can lead to migration of atoms [5]. Wei et al. [9] have studied CNTs under high current densities and their results show that CNTs did not fail for up to several weeks of testing and hence CNTs could be used potential candidates as interconnects for nanoelectronic devices. Thus, SWNT based network architectures on flexible media present a promising technology for Flextronics.

Applications of Flextronics place challenging demands on optical properties, dimensional stability, and solvent/moisture resistance of the substrates. Some of the commonly used polymers, including PEN [10] and PET [11], have been utilized as candidate flex-film substrates due to their acceptable properties. Another polymer, PDMS, which has a relatively low elastic modulus has also been used [12]. However, the difficulty of manufacturing a very thin layer, and compatibility issues with integrated circuit (IC) fabrication processes have limited its usage. Parylene-C, poly-para-xylylene-C, known to provide a very low permeability to moisture and gases, is the material of choice for coating critical electronic assemblies. Unlike PDMS which swells in most organic solvents [13], Parylene-C does not swell and is resistant to many solvents. In addition, parylene-C is relatively stiff (Elastic Modulus E = 3.2 GPa [14]) compared with PDMS (~0.75 MPa) [1]. Parylene-C has also high tensile strength (70 MPa) and yield strength 55 MPa [16] which helps easy removal from an attached surface without tearing. In addition, Parylene-C's compatibility with IC fabrication indicates that it can be a candidate, yet largely unexplored amongst others, as a flexible substrate. Normally, Parylene polymers are deposited at room temperature in a process similar to vacuum metallization, but at low vacuum conditions ($\sim 1.3\times10^{-4}$ atm). The mean-free-path during this process is on the order of 100 μm, which helps a conformal coating on the substrate in the Parylene deposition chamber. Several Parylene-C based devices have been made including sensors, actuators and probes [17-19].

FABRICATION

In this work, commercially available SWNTs supplied by Nantero, (Woburn, MA) were used to make lateral interconnect structures embedded between two Parylene-C layers. The resulting structure is shown in Fig. 1a. The electrical conductivity of these sandwich structures are then characterized under tensile loading. The fabrication process is illustrated in Fig. 1b and starts by depositing a thin (10 μm) Parylene-C layer on a 76.2 mm (3 in.) silicon wafer. Then, Au-electrodes (1500 Å) are patterned utilizing a transparency shadow mask. Next, a solution of SWNTs was drop cast and dried on top of a hot plate (90˚C). A 10 μm Parylene layer was next deposited to encapsulate the SWNT active bundles. The contact areas were then etched on the 2^{nd} Parylene-C layer, in order to expose the contact pads on the 1^{st} layer, with an inductively coupled plasma (ICP) etching tool using shadow mask patterning. After fabrication, the CNT-Parylene sandwich was peeled off from the silicon wafer.



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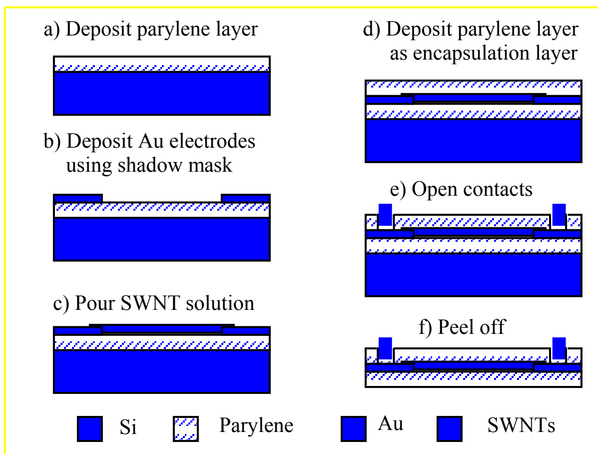

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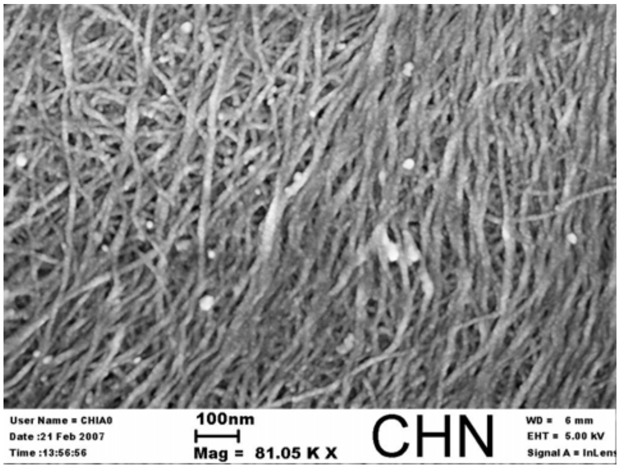
Figure 1 - (a) Optical photograph of the CNT-parylene sandwich-films (b) Fabrication process

The distribution of the CNTs on the Parylene was captured by an SEM image as shown in Fig. 2a. The drop casting process results in CNT bundles to dry on the surface in different orientations, and in an overlapping manner. It is likely that this overlapping in different orientations enabled continuous electrical conductivity in this thin flex film, even for large strain values, as discussed later.

The overall shape and dimensions of a typical sample tested for strain and electrical conductivity is shown in Fig. 2b, with test area of 2 mm × 10 mm. Since the entire sample surface was covered with SWNTs, it was crucial to reduce the strain near the electrodes. We designed to place the gold leads away from the test area, on the lower and upper horizontal corners of the specimen. These sections are further reinforced by gluing 250 μm thick cardboards on both sides (inset of Fig. 2c). The strain distribution in the specimen (Fig. 2c) shows that the horizontal sections experience approximately 1/1000th of the strain experienced in the test area, as determined by a finite element model (ANSYS, Canonsburg, PA) of the test structure.



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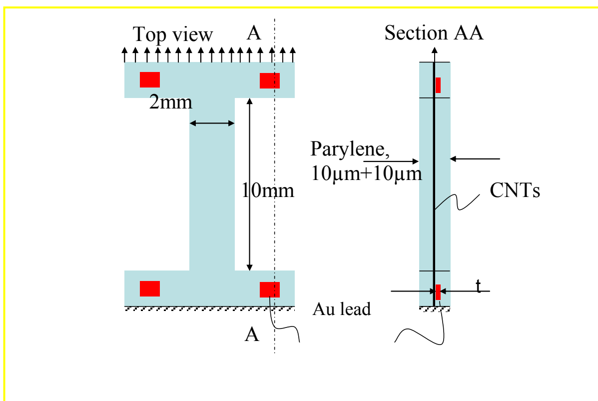

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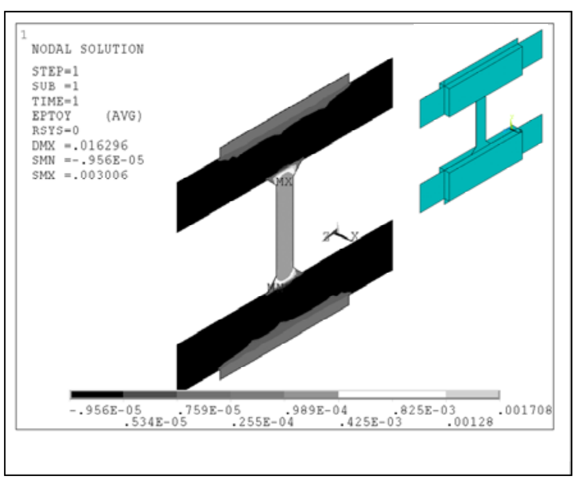


c)

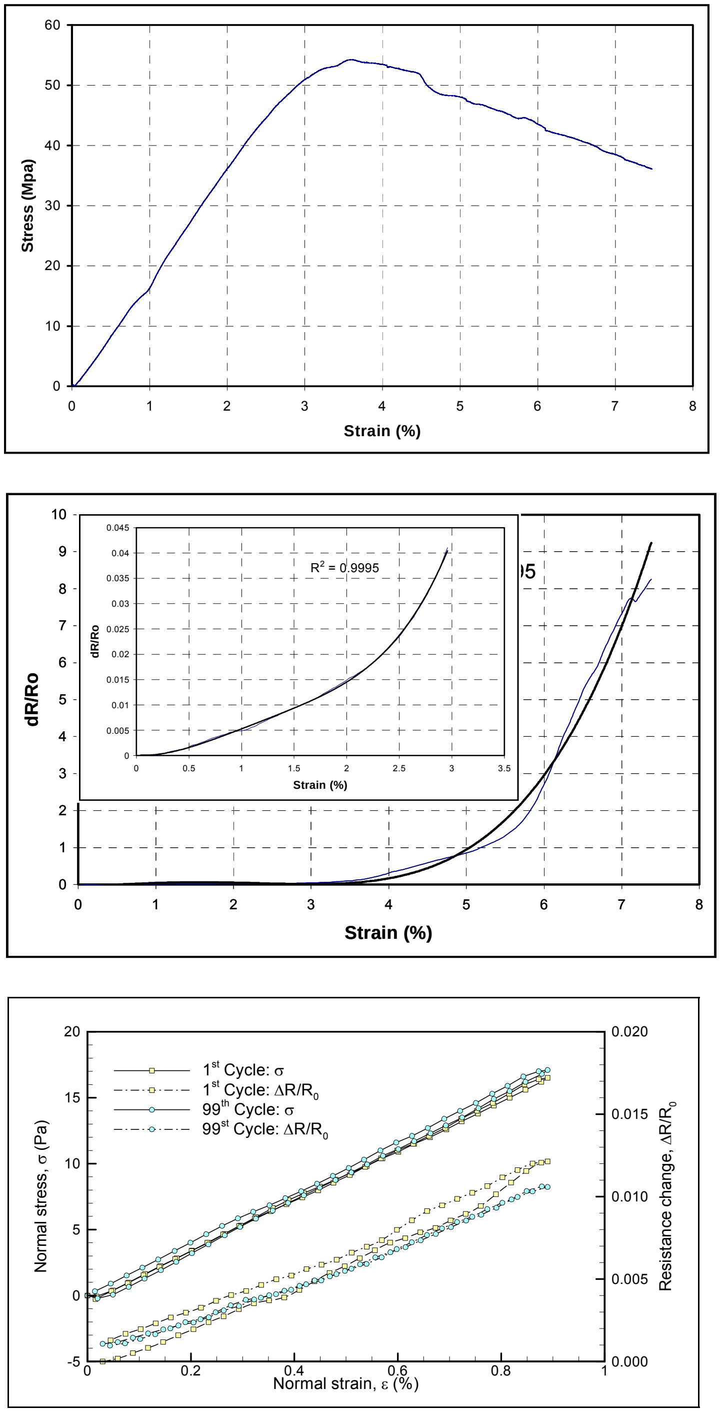
Figure 2 - (a) SEM image of the distribution of CNTs on the parylene substract (b) The tested dimension of sample (c) Strain distribution in the test specimen by FEA

TEST RESULTS

The test specimen is attached to a universal testing machine UMT-2 (CETR, Campbell, CA) for tensile testing. The resistance of the CNT layer is continuously measured during the test through a multimeter (Fluke, Model No 189 Everett, WA). Special attention is paid to ensure the grips hold the lower and upper horizontal sections without slip. A strain rate of 0.0033 mm/sec is applied to the specimen. The normal-stress (σ) is calculated based on the measured force and the cross sectional area (40×10^{-9} m^2). The normal strain (ε) is calculated based on the gauge length of 10 mm and the imposed elongation. The history of normal force, elongation, and electrical resistance are recorded during the tests. During the tensile testing the CNT-Parylene sandwich-film showed a linear-elastic response until a normal-strain value of $\varepsilon \cong 4\%$; the material failed at $\varepsilon_{Fail} \cong 7.5$ % strain as shown in Fig. 3a. The modulus of elasticity *E* of CNT-Parylene sandwich-film, based on a three sample study, was seen to be 1.93 GPa with a standard deviation of 0.08 GPa. The same parameter for 20 µm thick Parylene film was measured, as 1.46 GPa with a standard deviation of 0.22 GPa.

Fig. 3a shows the stress-strain curve, and Fig. 3b shows the relative change in resistance $\Delta R/R_0$, during this test as a function of strain. Initial resistance of the CNT-Parylene sandwich-film was $R_0 = 66$ Ohms. The resistance of the film increased 9 fold until failure. The sample was still conductive when the device failed. The $\Delta R/R_0$ shows a cubic-polynomial dependence on ε ($R^2 = 0.9995$) up to 7% strain. The details of 0 – 3% strain-range are plotted on the inset of Fig. 3b. This figure shows that the rate of change of resistance, $d\Delta R/d\varepsilon$, is fairly uniform until 2% strain, where $\Delta R/R_0 = 1.5\%$. The rate of resistance change increases thereafter, and at 3% strain the $\Delta R/R_0$ becomes 4%.

For load-unload tests, a specimen was strained to 1.3% and relaxed to 0% for four consecutive times. Then it was strained to 1.5%, 1.8% and 2% strain, and each strain level was repeated four times. Generally, a very small change in resistance (~1%) was observed when applying the strain range from 0% to 2% and the results were repeatable, with negligible hysteresis. A specimen was tested for 100 load-unload cycles in the 0 – 1% strain range. Figure 3 (c) shows the normal stress σ and the change in resistance $\Delta R/R_0$ in the specimen as a function of normal ε strain, for the 1st and the 99th cycles. While the σ -ε curve shows a very small amount of hysteresis for the 1st-cycle of loading, the 99th cycle is nearly hysterisis free. The same observation applies to the ($\Delta R/R_0$)-vs-ε behavior during the test. The film was still functional at the end of 100 cycles, where we stopped testing.



a)

b)

c)

Figure 3 - (a) Stress-strain test of 20μm CNT-parylene film (b) Change in resistance (c) Normal stress and strain and the change in resistance for the 1st and 99th cycles

SUMMARY AND CONCLUSIONS

We fabricated a CNT-Parylene sandwich-film, and characterized its mechanical and electrical properties. The macro scale samples (20 µm × 2 mm × 10 mm) were subjected to upto 99 cycles of load-unload tensile tests. Change of electrical resistance of the samples was monitored as a function of normal strain. Load-unload tests in the elastic range of the Parylene showed small resistance change (~1%) and negligible hysteresis in the R-ε data. Test to failure showed that the sample was conductive up to the failure strain of 7.5%. Furthermore, the resistance increased 9 fold during the failure test and the R-ε has a good fit by a cubic polynomial. Single-walled carbon nanotubes (SWNTs) embedded in Parylene-C layers remains conductive for reasonably high strain levels. The work presented here opens up the possibility of the use of CNT-Parylene films as interconnects in Flextronics and strain sensing elements in applications where biocompatibility and conformability (of Parylene) are important.

ACKNOWLEDGEMENTS

The authors would like to thank the staff and facilities of the Kostas Nanomanufacturing Center at Northeastern University (NEU). This research is funded by the Office of the Provost at NEU, and in part by NSF (NSEC) Center for High-rate Nanomanufacturing (Award # NSF-0425826).

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Manufacturing and Performance Evaluation of Organized Carbon Nanotube-Parylene Multi-Functional Active Thin-Films	**C.-L. Chen, E. Lopez, M. Dokmeci, Y. J. Jung & S. Müftü,** Northeastern University, USA

Name & Affiliation	**Question**
K. Reid, Oklahoma State University	Do you attempt to align the carbon nanotubes?
Name & Affiliation	**Answer**
E. Lopez, Northeastern University	That will be the next step. My colleagues are working on different processes to grow the carbon nanotubes either vertically or in lines.
Name & Affiliation	**Question**
K. Reid, Oklahoma State University	What is expected to be the gain from that?
Name & Affiliation	**Answer**
E. Lopez, Northeastern University	Using or casting method resulted in random orientation. We could pull in different directions and still maintain that conductance. If we align the carbon nanotubes there could be more chemical bonds.
Name & Affiliation	**Question**
Dan Carlson, 3M	Was the parylene to provide a protective layer?
Name & Affiliation	**Answer**
E. Lopez, Northeastern University	The idea was that these would be used as flexible electronics, so the electronics are carried within the parylene itself.
Name & Affiliation	**Question**
Dan Carlson, 3M	What is of the cost for the nanotubes and the parylene relative to other materials that might be used for this application?
Name & Affiliation	**Answer**
E. Lopez, Northeastern University	I'm not sure.

DISCUSSION II

Leaders: *A. Thuer, Avery Dennison, and B. Feiertag, Oklahoma State University*

Name & Affiliation	**Question**
Neil Michal, Kimberly Clark	Dan Carlson's talk this morning was fascinating. When do you know if you need the high performance controls and where is an independent source available to come in and give you an assessment? Typically we call a vendor who is many times motivated by selling equipment versus giving us an independent eye on what makes sense or what you can do to tune up what you've got versus you should really try something different.
Name & Affiliation	**Answer**
Dan Carlson, 3M Company	The only answer I can offer is to employ advanced controls when the traditional control methods do not work. That is the message I was trying to convey is that I have always fought advanced controls because people reach for them too quickly. They haven't done all the other fundamental things first – fix the plant, get the fundamentals as good as possible. After the fundamentals have been taken care of and traditional controls methods have failed then I look to the advanced control methods. Again, the additional complexity, that inevitably we have these unanticipated areas of operation make advanced controls more challenging. Is there a simple way to say I need advanced controls? The only good answer I can offer is when you have exhausted all your available experts and they have done the best job they can and then you still can't solve the problem or get the performance you like.
Name & Affiliation	**Comment**
Neil Michal, Kimberly Clark	The challenge is that we have fewer and fewer technical representatives and sometimes we have to go out and ask for help.
Name & Affiliation	**Comment**
Dan Carlson, 3M Company	Look at all the IWEB papers on the subject of controls; there's been lots of great controls work. But there is a disconnect between the theory and the application. The trouble we have, being 3M, is that I want to purchase solutions; I don't want to have to develop them. The only time I develop solutions is when we can't find a reasonable solution. The trouble is, in the present form the advanced control solutions are too complex, or not in a form suitable for a manufacturing environment. That's where I believe the big disconnect is. That's why I want to encourage more partnerships between the universities and others so we can see some of these methods in a form that we can apply in a manufacturing environment.
Name & Affiliation	**Question**
Dave Roisum, Finishing	Instead of where should we use advance controls, where do

Technologies	we see them in web handling? This would be on commercial applications, things like tension control guiding, etc… Do we have any products out there that we are aware of that have advanced controls?
Name & Affiliation	**Answer**
Dan Carlson, 3M Company	There are products, like in the Rockwell line. There's the Control Logic platform which is a generic controller and you can design it yourself. That is one of the issues you have, you're not only trying to solve the control problem but you are trying to design a controller. The other product I hinted was developed by Krebs. Krebs presented a paper at the Fourth IWEB. The strategy Krebs used, it wasn't advanced control, in a sense it was conventional PID. What he did was implement a configurable strategy that allowed the user to implement these things and he well documented his strategy. In this approach you're leveraging a control expert's knowledge and you have confidence that this controller has been implemented correctly. You're just configuring, tailoring that controller to the application. That is the type of products I would like to see more of. They are encapsulating this expertise and letting the less sophisticated user apply them to their applications.
Name & Affiliation	**Answer**
Mark Weaver, Rockwell Automation	I support Dan's contention about advanced controls. We have incorporated, for instance, features in our products that would be considered as advanced. We don't see them in competition products. We tend to take the approach that Dan does. We're not going to apply those features unless we have exhausted all conventional opportunities. The reason we do that is we try and look at what the application requires and we try to avoid having to have an application dependent upon an individual or a company. The control system should be reliable enough that it's competent to control 80% of the time in the regime you need to produce. Where we see needs for this is where we have extraordinary mechanical problems. A lot of our controls are used to try to compensate for poor mechanics such as resonances and backlash. The nemesis of good control is lost motion. We have ways we deal with that, but the real solution is to eliminate the poor mechanics issues in the design stage. That is, I think, the better way to go. When you can't do that, our controllers and other people's controllers can provide solutions and I know Dan has probably dealt with many of those.
Name & Affiliation	**Answer**
Keith Good, Oklahoma State University	I have a servo hydraulic Instron material testing system and we test radial modulus properties of various kinds of materials. Maybe at one point, you are compressing a pretty stiff stack of polyester and things are going pretty well and then next you decide to compress a stack of non-

woven. The machine starts howling and going completely out of control and you reach down to the Instron control panel and there's this little button that says adaptive control and you hit it and within seconds you are in control and the problem is gone. The Instron is automatically adjusting P, I and D until it knows it has found a satisfactory solution and you are back in control. In response to Neal Michal's question on when and how do we employ advanced controls. I think some of our challenges come from machines that have to run multiple products that have varied web widths and thicknesses, varied modulus, and various tension levels. If you are running converting machines where you have to change product x times a day, maybe we get to a point where adaptive control makes sense, where you don't expect the operator to have to keep up with good P, I, and D parameters for every web the machine has to run.

Name & Affiliation	**Question**
Bob Lucas, Winder Science	The question is when do you have the need for the highly sophisticated control systems. In the world I have been living in, you may not have the opportunity to apply these high technological methods. The equipment we are trying to control has so many deficiencies. An example is when you have pneumatically actuated brakes controlling web tension in low budget converting plants that can't afford the more exotic control systems. Some of the techniques that we resort to would probably make the control engineer shudder because we break all the rules. Sometimes I even eliminate a classic PID controller and I use a totally different approach because I can come up a very simple functional thing that works and is able to handle the pneumatic transport delay issues. I end up with a very functional system without getting involved with dynamically solving matrix solutions to a control problem. Sometimes the solution comes from solving the right problem and keeping it simple.

Name & Affiliation	**Comment**
P. Pagilla, Oklahoma State University	Regarding the advanced control; the conventional wisdom of this group has been to exhaust all other possibilities and adopt advance control only when that is needed. But we don't know when it is needed. When we consider other industries, for instance I have dealt with the aerospace industry; they are not taking that approach. They are employing advanced controls; they are looking for possibilities to improve. They are not sticking to their old PID controller algorithms. They are taking a totally different approach. What I see in the web handling community is the hiring practice of people with advanced control knowledge. We don't see many experts or people who have had more

Name & Affiliation	Comment
	modern control approach backgrounds joining these companies. Whereas if you go to Honeywell or Seagate or Applied Materials, they are always hiring advanced control experts. My students are being hired by companies like Caterpillar and Boeing who are employing advanced control algorithms. There has been resistance to advanced controls in the web handling community because we don't understand; we don't even want to try it. I think this is exactly opposite to what Pete Dulcamara said in his keynote speach. There is this resistance to try advanced controls. Everyone wants better performance, but if PID is running fine, let's stick to it, let's try fixing other problems.
Name & Affiliation	**Comment**
Jerry Brown, Essex Systems	As a controls engineer, I think to do anything with advanced controls, you have to have an advanced company. I was in a plant the other day where they had a pair of guys that had really gotten good at wonder-wear. They had a line with over a dozen tension zones. They had a situation where they had dozens of recipes for a variety of products. They were having a terrible time. They were asking me for help and I didn't want to tell them it was these two guys that were their biggest problem. This machine had severe mechanical problems. They had an oven where the bearings in the rollers were all frozen. They had nip rolls on bearings that had a quarter inch of play. They had bowed rollers that didn't turn. The mechanics of the web line were a catastrophe. Ultimately, I had to tell them you have to overhaul the line. Maybe after they do they solve their mechanical problems they can make PID tension control work.
Name & Affiliation	**Comment**
Karl Reid, Oklahoma State University	Advanced controls are advanced depending on who you are and where you are. Modern controls, or most of what we consider as modern controls, were developed 20 years ago or before and have just been small changes since that time. Several years ago, again about 20 years ago, the aerospace industry was grappling with the same question. There was an independent authority; someone in the Air Force came up with a paradigm problem. He said, ok, all you guys that have the fancy control strategies attack this problem and there was a fly off. It was an incredible thing. They brought it to the American Control Conference, at that time it was called the Joint Automatic Controls Conference, of all the societies that have control engineering interest. So, it may be time for us to develop paradigm problems in web processing machinery and consider some real problems. Maybe we have a panel or have a group of people challenged to address those problems, such as Keith mentioned, where a machine becomes unstable after changes of operating conditions or

Name & Affiliation	Comment
	parameters with the material you are processing. Maybe it's time to do something like that.
Name & Affiliation	**Comment**
Dan Carlson, 3M	I don't disagree with what Prabhakar Pagilla said. The distinction is the application of advanced controls should be limited to high value problems. If you have a high value application that really needs the performance, the concern I'm expressing is that too often they want me to patch up a machine that should really be fixed with mechanics and not advanced controls. It is a misapplication. Another thing that happens is, if I implement advanced control before the application is ready, then I spend my time supporting it because it is not in a form supportable by someone else and then I can't go on to other things. So, that's the perspective I'm offering and that is what I am asking for – if it is in a commercial controller and then we apply it and the plant is not relying on me and they're relying on the manufacturer and it's 24/7 support, in that environment it's really important to have multiple sources of support. That's what I'm trying to encourage here, is to get more of those advanced control things into commercial equipment so we can take advantage of it. They will bring great benefit, but if it's done too early, people will get a bad taste in their mouth. It will work fine, but it's like that Airbus crash – it took a long time to get past that disaster because they didn't get all those things addressed out of the gate. That's the message I'm trying to convey. I'm not saying we shouldn't use advanced control, just when it's appropriate and when it's applied to the high value problems.
Name & Affiliation	**Comment**
J.J. Shelton, Oklahoma State University	I was talking about backlash earlier, and to elaborate on that, it is appalling, again I have to apologize beforehand if I'm putting words in Prabakhar's mouth, but he and I have discussed the ignorance that gets into print. He has said that half the papers on backlash are also calling it dead band. Backlash is destabilizing, dead band is stabilizing. Dead band, of course, contributes to error. But, I thought that everybody that had any experience with controls would know the difference between dead band and backlash. If they don't there is really no hope. I guess I will just comment twice a day on backlash until someone gets tired of listening to me and to learn if they don't already know the difference.

Name & Affiliation	Comment
Unknown	I went through a learning experience recently with backlash. I was trying to simulate it with Simulink. I went and selected the block that said backlash on it and it wasn't behaving how I thought it should. So, I went back to first principles, I pulled out some work that Prabakhar and John had done and looked at it and you know what? The block in Simulink that calls itself backlash is not the backlash that we need so be careful. Go back to first principles and look what's really happening. So, that is my comment on when we start simulating backlash. It's important to really understand what you mean. You're talking about lost motion, where you're essentially uncoupled. A good way to ruin your tension control is to introduce a lot of backlash in the system. You must minimize it. You can't control a system that's not connected.

Name & Affiliation	Comment
J. J. Shelton, Oklahoma State University	I wanted to relate an experience that most people should avoid because it costs money. This was a fairly big web guide and there was backlash in the cylinders connecting to an unwind stand. If it is a clevise mounted cylinder it will have backlash, and this was a clevise mounted cylinder. There were other problems, but we got them fixed and had a serious backlash problem. The solution was connecting two cylinders, push-push instead of push-pull. This was inexpensive in comparison to the rest of the equipment. For motor controls, whether it's a web guide or tension control system, I visualize two motors pushing against each other in the same way if you can't fundamentally get rid of the backlash in the system.