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Feasibility study to evaluate candidate materials of nanofilled block copolymers for use in ultra high density pulsed power capacitors

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Composite dielectrics offer opportunities to combine the high permittivity of inorganic fillers with the high breakdoen strength (Ebd) of polymer matrix to obtain high energy density capacitors. Our strategy of grafting block copolymer (BCP) to nanoparticles (BCP-g-NPs) to chemically match the corona of NPs with BCP matrix has resulted in a highly dispersed BCP system without compromising the original order of BCP. We performed relative permittivity measurements of Bcp-g-NP filled BCP composite and showed enhancement over unfilled bcp on aluminum coated quartz substrate. We successfully demonstrated a novel approach to multilayered electrostatic capacitor films showing the first-ever application of self-assembling BCPs as the nanostructured dielectric media in electrostatic capacitors. Using controlled ordering of lamellae-forming BCPs via cold zone annealing soft-shear strategy of directed self-assembly, highly ordered lamellar architectures were obtained in micron thick BCP films, yielding up to a ~50% increase in Ebd compared to as-cast BCP films. Since energy storage density has a quadratic dependence on the EBD, significant enhancement in energy density was achieved by using nanostructured BCPs.

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Feasibility study to evaluate candidate materials of nanofilled block copolymers for use in ultra high density pulsed power capacitors

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Project Summary :

Composite dielectrics offer a unique opportunity to combine the high ε of inorganic fillers with the high V_{bd} of a polymer matrix so as to formulate high energy density capacitors. We use directed self-assembly of block copolymers (BCP) to rapidly fabricate highly aligned BCP-TiO2 composite nanostructures in thin films under dynamic thermal gradient field to synergistically combine the high ε of functionalized TiO2 and high V_{bd} of BCP matrix. Our strategy of grafting BCP to nanoparticles in order to chemically match the corona of nanoparticles with BCP matrix has resulted in a highly dispersed BCP system without compromising the original order of BCP. We also have performed relative permittivity measurements of ~20 wt% bcp-g-np filled bcp composite and it showed an enhancement compared to the unfilled bcp on aluminum coated quartz substrates.

Our project has also successfully demonstrated a novel approach to multilayered electrostatic capacitor films showing the first-ever application of self-assembling BCPs as the nanostructured dielectric media in electrostatic capacitors. Using controlled ordering of lamellae-forming BCPs via our cold zone annealing soft-shear (CZA-SS) strategy of directed self-assembly, we have shown that highly ordered lamellar architectures can be obtained in micron thick BCP films, which yield up to a ~50% increase in breakdown strength (E_{BD}) when compared to as-cast BCP films with no internal structuring We attribute this enhancement to the “barrier effect” of the multilayered structure, which forestalls the electrical breakdown pathway and causes the film to fail at higher voltages. Since energy storage density has a quadratic dependence on the E_{BD}, significant enhancement in energy density was achieved by using nanostructured BCPs.

Research Accomplishments

Among the various energy storage technologies such as batteries, fuel cells and capacitors, electrostatic or solid-state capacitors possess the advantage of high power density due to relatively fast energy storage and discharge capabilities. However, the energy storage density of these capacitors is limited by the dielectric properties of the materials that separate the oppositely charged electrodes, falling significantly short of rising demand in advanced applications. There is a critical need to develop not only new dielectric materials, but also to control their morphologies at the nanoscale in ways that can significantly increase their energy storage capacity thereby reducing the size, weight, and cost of electronic devices and power systems.

Fundamentally, the dielectric material limits capacitor performance, with the energy density governed by the product of dielectric permittivity (ξ) and breakdown strength (V_{BD}). Therefore, further improvements in capacitor energy density are greatly reliant on improving either permittivity, breakdown strength or finding improvements through a combination of both properties. Recent attention has been focused on polymer nanocomposites, composed of fillers with high permittivity and polymer matrices with high breakdown strength, to engineer the dielectric properties, integrating complementary elements of permittivity and breakdown strength for an enhancement in energy density. However, while improvements in permittivity through higher nanoparticle loading are readily achievable, enhancements in the breakdown strength have been difficult to obtain. The interfacial bonding between functionalized nanoparticles and the polymer matrix is one of the key paths to improved voltage endurance and enhancement in the break down strength. Others have noted that interfacial tailoring, either of the nanoparticle surfaces to improve dispersion, preventing high field stresses due to material imperfections, or macroscopic layering of alternate polymers providing a barrier to the propagation of electrical breakdown, can both positively impact the breakdown strength. However, a fundamental understanding of these phenomena is required to fully taken advantage of these disparate ideas.

To investigate this disparate ideas, initially we considered symmetric diblock copolymers like Poly (styrene-block-methyl methacrylate) (PS-b-PMMA)) that micro- phase separate at the nanoscale to give a well-ordered parallel lamellar structure under the right annealing conditions as shown in **Figure 1** below. Such a structure should provide multiples interfacial barriers and impedes the electronic breakdown pathway by providing high tortuosity, thereby increasing voltage breakdown strength.

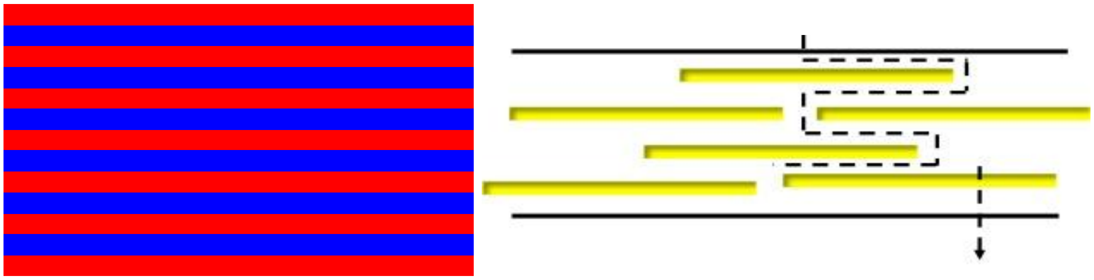


Figure 1. Schematic of a well-ordered lamellar block copolymer providing path tortuosity to electrical breakdown.

1) High-Barrier Effect in Cold Zone Annealed Soft-Sheared Neat Lamellar BCP System

We have confirmed that highly oriented BCP morphology can play an extremely important role in voltage breakdown characteristics. Specifically, ordered lamellae morphology was found to have higher E_{BD} compared to cylinder forming BCP, directly confirming the role of interfacial geometry on electrical "treeing breakdown" effect on E_{BD} for the first time. **Figure 2** shows a dynamic thermal gradient-based soft-shear method used to direct the alignment of lamellae-forming BCP films to create a highly parallel ordered, layered architecture. The film is nearly 0.7 µm, for which oven annealing only produces mixed morphology in the film interior. This is also a significant achievement because the energy stored scales with *squared power of* E_{BD}. Furthermore, our studies have demonstrated that E_{BD} scales directly with domain spacing of BCP lamellae. Low Mw (below/near entanglement) BCP fails at lower fields than high Mw (highly-entangled) BCP of similar composition. We have also established that a PS-PMMA BCP with perpendicular lamellae showed the lowest breakdown ($E_{BD} \sim$ 200-300 V/µm) even lower than the random morphology as-cast film. In contrast, its parallel BCP lamellae film version showed the highest breakdown ($E_{BD} \sim$ 500+ V/µm) even higher than the constituent polymer blocks (PS~250 V/µm and PMMA~500 V/µm). This can be attributed to the role of interfacial geometry on electrical "treeing breakdown" effect in BCP system. However, ordering high Mw systems (with thermal annealing, e.g. CZA-SS) remains a challenge due to sluggish kinetics of ordering at modest temperatures.

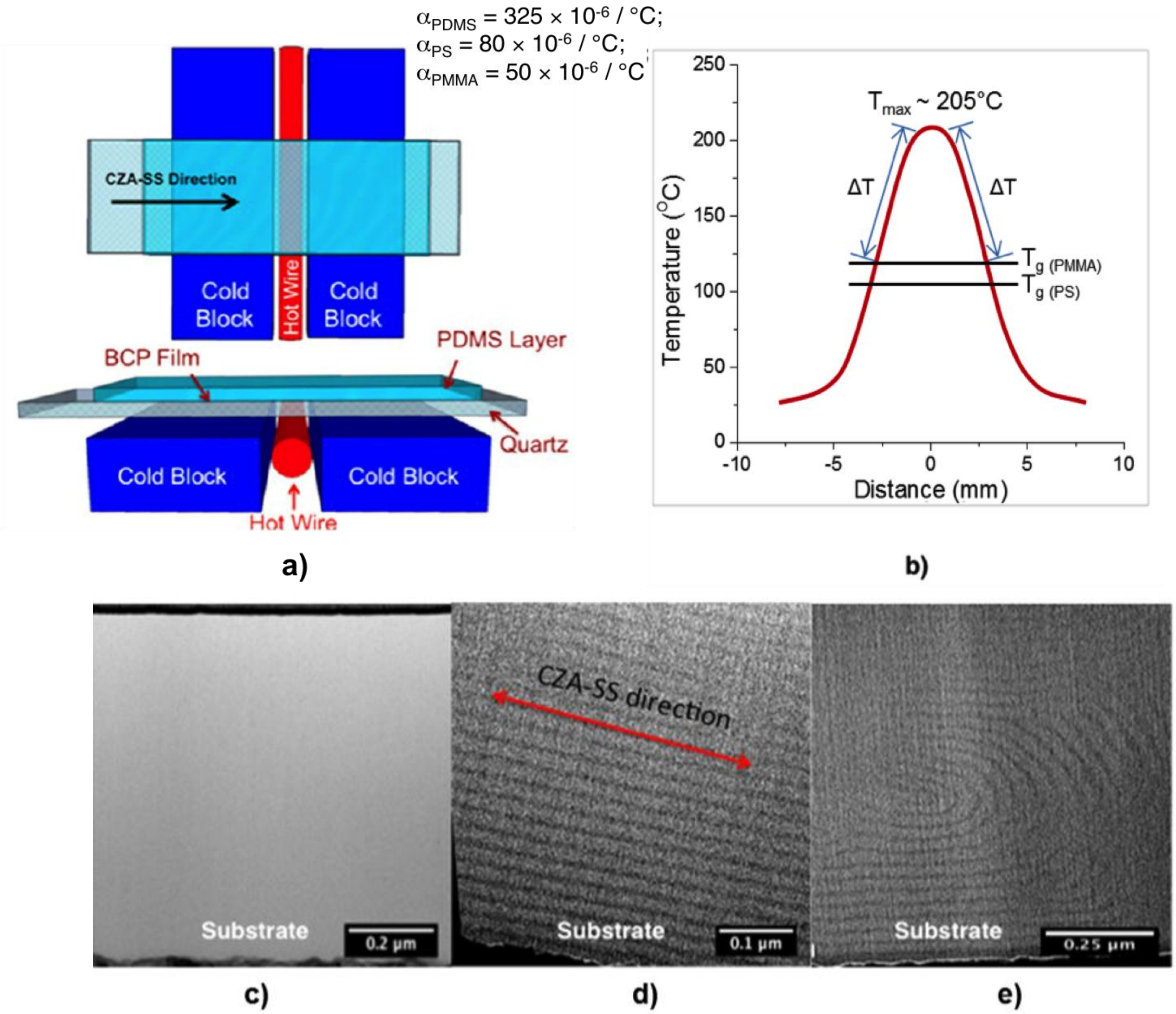


Figure.2. A dynamic thermal gradient-based soft-shear method used to direct the alignment of lamellae-forming BCP films to create a highly ordered, layered architecture resulting in ~50% enhancement in the E_{BD} over unstructured films (work being submitted to Nature communication). **a)** Schematic for directed self-assembly of BCP film using Cold Zone Annealing-Soft Shear (CZA-SS). **b)** Sharp thermal gradient profile induced by CZA setup and linear thermal expansion coefficients of materials. Cross-sectional TEM images (no stain) of PS-*b*-PMMA (19.5-*b*-18.1 kg mol^{-1}) film on Al substrate: **c)** as cast, **d)** CZA-SS at $T_{\max}$ = 205 ºC, V = 5 μm/s, and **e)** oven annealed for 96 hours at 205^{0}C under vacuum.

Table 1 summarizes the breakdown information for the different processing conditions according to Weibul plots analysis. Permittivity measurements of various BCP configuration showed that parallel lamellae and cylinder configuration have better β factor in Weibull plot than 'as-cast' or other high morphologies and orientation. The β factor is a measure of the onset of breakdown relative to $E_{BD,}$ so a high β factor is interpreted as a capacitor with low defect density. BCP morphology with nanoscale layering was found to have a high β factor due to the lowered probability of defects occurring within a certain volume in multiple layers. Annealing of BCP films further improves the β factor in both lamellae and cylinder forming BCP films. It is quite apparent that the CZA-SS processed BCP films have the highest breakdown strength with reasonable β values! This study has provided a simple but elegant approach to multilayered morphology while providing the highest breakdown strength of neat BCP films. The findings of the work is being submitted for peer-reviewed publication.

Table 1. Summary of characteristic dielectric breakdown strength (E_{BD}) and shape parameter β measured for PS, PMMA, and PS-*b*-PMMA films.

Sample		E_{BD} (V/μm)	**b**
100k PS		251	7.1
88k PMMA		494	10.2
19.5k-b-18.1k PS-b-PMMA	6 h oven	289	4.1
	As cast	346	5.4
	96 h oven	467	6.4
	CZA-SS	520	4.6
33k-b-33k PS-b-PMMA	6 h oven	316	6.4
	As cast	403	5.8
	96 h oven	529	6.2
	CZA-SS	599	6.1

3) Functionalization of TiO_2 nanoparticles with BCP

Introducing high permittivity nanoparticles into bulk polymer films can enhance the energy storage properties; however, reduced breakdown strength of these polymer nanocomposite films remains a challenge. Field exclusion, driven by permittivity mismatching, and agglomeration are commonly cited for triggering premature breakdown. Shifting focus from homopolymers to block copolymers may potentially assist in nanoparticle dispersion. Since block copolymers microphase separate to form highly ordered structures, they provide a template for controlling nanoparticle location. Furthermore, the interfacial barriers formed by these blocks can force the propagating electric field to travel along a more tortuous path. These morphological improvements would prevent the precipitous decline in breakdown strength previously observed in polymer nanocomposites.

Presently, there is very little knowledge as regards the dispersion and orientation of nanoparticles grafted to BCPs, and even less on how the size and the type of nanoparticles affects their dispersion state in a BCP matrix. BCP morphologies such as lamellar, cylindrical, or spherical, can form ordered arrays of layers, lines or spots respectively, with feature dimensions of 10–50 nm. While these ordered arrays can serve as templates for sequestering the tethered nanoparticles, fundamental questions remain as to the localization of the NP-g-BCP within a BCP block or at interface and how directed self-assembly may be used to drive a certain organization of the NP-g-BCP relative to the BCP matrix ordered domain.

Dispersion of nanoparticles in the BCP matrix is intimately related to the underlying interaction of host polymer and ligands present on the surface of the nanoparticles. Surface functionalization of inorganic nanoparticles is widely used to improve the dispersability of nanoparticles in solvents and their compatibility with one of the components in block copolymer system (BCP). In particular, the interaction between the nanoparticles and polymer domains in BCP can be improved by grafting compatible polymer chains onto the nanoparticles. Two approaches are commonly used for grafting polymers onto inorganic nanoparticles (1) attaching monomers to the nanoparticles followed by the polymerization of the grafted monomer units, or (2) directly attaching pre-prepared polymer to the functional groups on the nanoparticle surface.

Our strategy of grafting BCP to TiO_2 nanoparticles was based on chemically matching the corona of nanoparticles with BCP matrix and evaluate the dispersability of BCP-TiO_2 system in

BCP matrix without compromising the morphology of BCP system. The reaction scheme for grafting TiO_2 nanoparticles with BCP using Williamson ether synthesis is shown in **Figure 3**. XPS and GPC of cleaved sample established that the surface of nanoparticle is indeed functionalized with the PMMA-b-PS. The functionalized nanoparticles showed improved thermal stability than pristine BCP which renders these functionalized nanoparticles suitable for thermal processing.

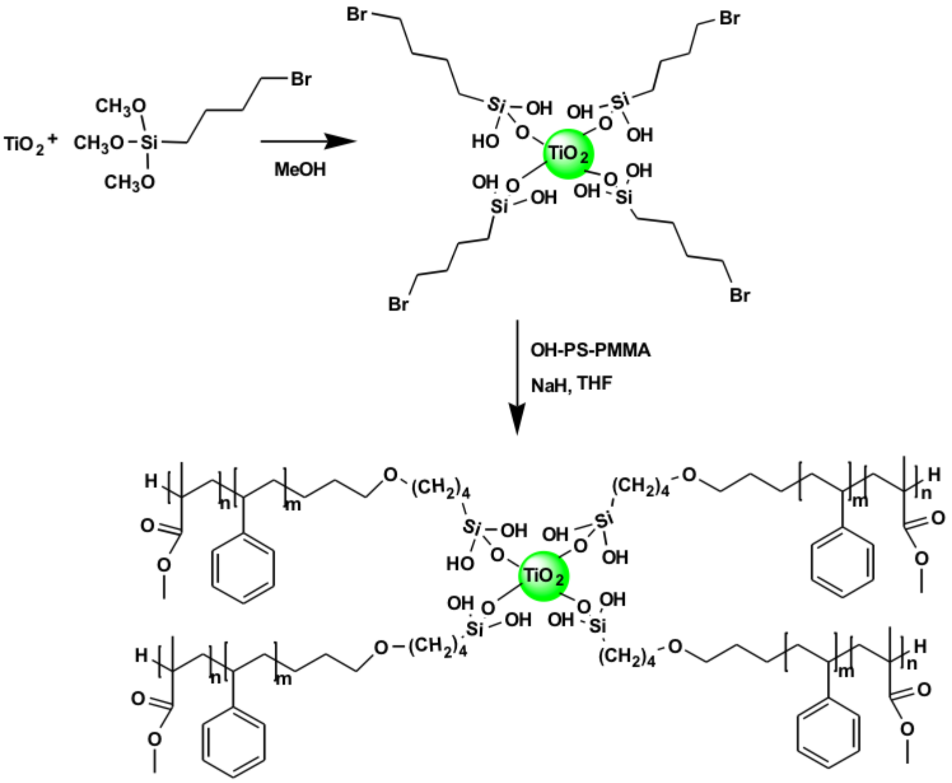
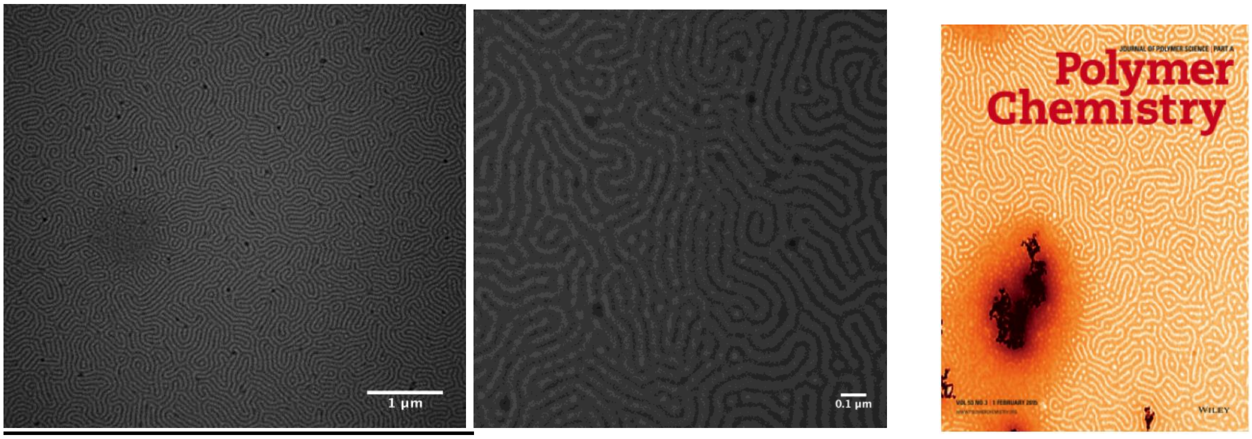


Figure 3. Functionalization of TiO_2 nanoparticles and the subsequent grafting of BCP, PMMA-b-PS.

4) Ordering and dispersion of polymer tethered nanoparticles in block copolymer films

We performed systematic comparative dispersion study of bcp-g-nps in homopolymer and BCP system and observed that nanoparticles dispersed well in BCP and PMMA matrix, but strongly aggregated in PS (Figure 4). Control dispersion study with pristine nanoparticles/BCP system showed that the pristine nanoparticles aggregate in BCP matrix (**Cover of Journal of Polymer Science: Polymer Chemistry Edition- Figure 4 inset)**. These results strongly suggest that dispersion of nanoparticles in BCP is facilitated by the asymmetric grafted "ligand" interactions. In the nanocomposite thin films, the bcp-g-nps are preferentially sequestered at the PMMA-b-PS interface with ligands spanning both the PS and PMMA domains in inverted fashion in the linear cylinder-forming PMMA-b-PS diblock copolymer matrix. Irrespective of the loading of nanoparticles, the order of the matrix block copolymer in nanocomposite film was retained as evidenced by the sharp ring indicative of isotropic structure in the GISAXS image. Our results highlight the role of functionalization of high permittivity nanoparticles such as TiO_2 as a strategy towards controlled dispersion of nanoparticle in a specific domain or at the interface for block copolymer based capacitors.



(a) (b) (c)

Fig.4. a) Lower magnification and b) higher magnification TEM (no stain) images of oven annealed composite films with 10 wt.% loading of BCP-g-TiO2 nanoparticles. The cover figure of our work shows the aggregation of pristine TiO_2 nanoparticles in a PMMA-*b*-PS block copolymer (BCP) thin film matrix.

Next, we performed CZA processing of composite films with 20 wt.% loading of BCP-g-TiO2 nanoparticles and unfilled PMMA-*b*-PS block copolymer (BCP) thin film matrix. With cold zone annealing–soft shear (CZA-SS) annealing, we successfully achieved alignment of bcp-g-nps along the BCP cylinders in the direction of CZA-SS. In collaboration with WPAFB scientists, we also have performed relative permittivity measurements of ~20 wt% bcp-g-np filled bcp composite and it showed an enhancement compared to the unfilled bcp on aluminum coated quartz substrates.

5) Effect of Thermal Processing Conditions on the Morphology of NPs loaded BCP Film

Additionally, we have recently shown that the BCP morphology can be influenced by the addition of nanoparticles in BCP system and can be flipped depending upon the processing conditions (**Figure 5**.). This gives additional parameter other than ligand chemistry in tuning the BCP morphology. Previously we have shown that low levels (1-3) wt% of gold and fulleropyrrolidine (f-np) nanoparticle addition can induce synergistic orientation effects on BCP film structure, switching PMMA cylinders from vertical to parallel orientation in CZA-S processed nanocomposite PS-b-PMMA films, not observed in counterpart oven annealed BCP films. For the first time, it has been demonstrated that the orientation flipping can be reversed in a nanoparticle block copolymer system where the nanoparticles are apparently well-dispersed. [**Figure 5 inset- Cover of Journal of Polymer Science : Polymer Physics, Edition]**. We anticipated a biased selective sequestering of these nanoparticles that will strongly depend on BCP-g-NP concentration. We anticipate that as the concentration of BCP-g-NP increases in the BCP matrix, the self-assembly properties of the BCP-g-NP can start to compete or even dominate the ordering morphology.

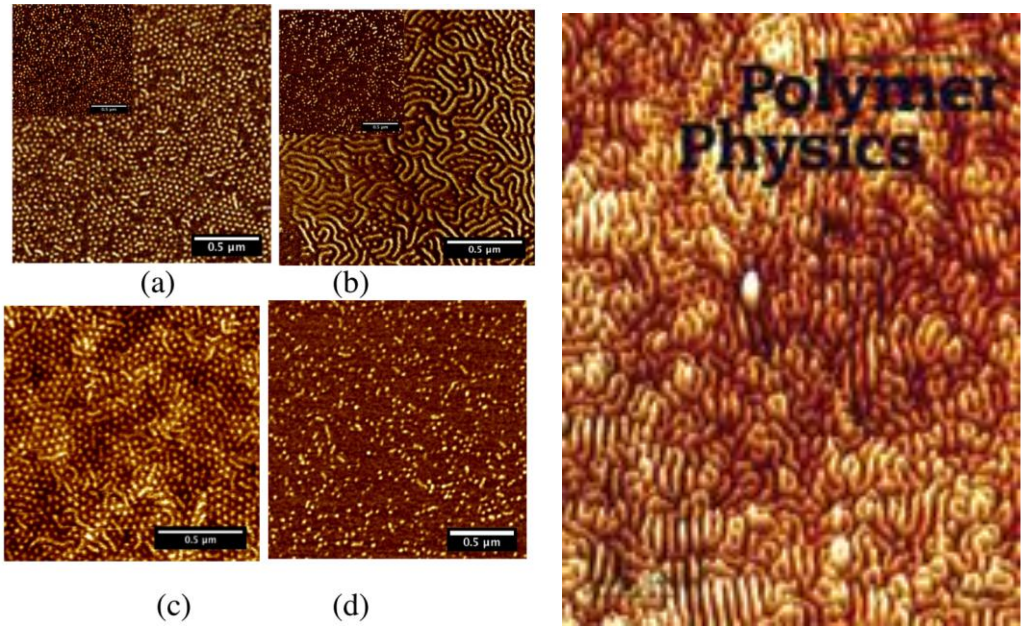


Figure 5. AFM surface topography phase images of PS-b-PMMA-fCdS composite films zone annealed at T_{max}=205^{0}C, V=5 μm/s with a) 0% & b) 5% nanoparticle concentration, and V=10

μm/s with c) 0% and d) 5% nanoparticle concentration. Inset: AFM phase images of corresponding oven annealed samples (Scale bar: 0.5um) Our work appeared in the Cover of J Poly. Sci. Polym Phys. Edn.

Conclusions:

We have made substantial progress in the effort to develop BCP-based capacitor materials, modify the surface chemistry of nanoparticles, influence the dispersion of nanoparticles in BCP matrix and control the sequestration of nanoparticles in BCP. A significant achievement of our work is the use of grafting-to approach to tether PS-PMMA BCP chains on to TiO_2 nanoparticles and also controlling BCP orientation through NP loading and CZA processing. The findings of this study have appeared on the *Cover* of Journal of Polymer Science: Polymer Chemistry, as well as on the *Cover* of Journal of Polymer Science: Polymer Physics. We observed that BCP morphology plays a vital role in breakdown voltage (E_{BD}) characteristics of matrix. Specifically ordered lamellar morphologies were found to have higher E_{BD} compared to cylinder forming BCP, confirming the role of interfacial barrier effect in electrical "treeing breakdown" on E_{BD}. This is a significant achievement because the energy storage density scales with squared power of E_{BD}. Finally, we noted that BCP with perpendicular lamellae showed the lowest breakdown followed by 'as cast' BCP films and the highest values were obtained for parallel BCP lamellae films. This work is in the process of submission to peer-reviewed journal. This study can be fundamental to design of novel capacitors. With the potential to synthesize new BCPs having optimum dielectric properties and applying directed self-assembly methods, it is possible to obtain a catalog of tailored BCP materials that can be used to fabricate high-energy density, pulsed power, flexible capacitor films for use in directed energy, electric vehicles and future flexible electronics.

Archival publications (published) during reporting period:

1. P. Pitliya, J. Chapa, G. Singh, A. Karim, and D. Raghavan, *Polymer*, **54**(4), 2013, 1415-1424.

2. K. A. Masser, H. Yuan, A. Karim, and C. R. Snyder, *Macromolecules*, **46**, 2235-2240(2013).
3. Hongyi Yuan, Gurpreet Singh, Dharmaraj Raghavan, Abdullah M. Al-Enizi, Ahmed Elzatahry, and Alamgir Karim, *ACS Applied Materials & Interfaces,* 2014 *6* (16), 13378-13388

4. Shimelis T. Hailu, Saumil Samant, Christopher Grabowski, Michael Durstock, Alamgir Karim, and Dharmaraj Raghavan, *Journal of Polymer Science Part A: Polymer Chemistry*, 2015, 53(3), 468–478.

5. S. Samant, S. T. Hailu, A. M. Al-Enizi, A. Karim and D. Raghavan, *Journal of Polymer Science : Polymer Physics Edition*, 2015, 53(8) 604-614.

6. Saumil Samant, Christopher A. Grabowski, Kim Kisslinger, Kevin G. Yager, Guangcui Yuan, Sushil Satija, Dharmaraj Raghavan, Michael F. Durstock, Alamgir Karim, Electrostatic Capacitor Design Using Highly Ordered Lamellar Block Copolymer Films, *Nature Communication* in the process of submission (2015).

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Abstract

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