## **Experimental report**

Proposal: CF	G-2649			Council: 4/20	19	
Title: Stu	Studying the percolation of soft and rigid latex nanoparticles duringannealing					
Research area: Phy	vsics					
This proposal is a nev	r proposal					
Main proposer:	Calvin BRETT					
Experimental team:	n: Calvin BRETT					
	Stephan ROTH	Stephan ROTH				
	Alexandros ALEXAK	Alexandros ALEXAKIS				
	Andrei CHUMAKOV					
Local contacts:	Alexei VOROBIEV					
Samples: Latex						
Instrument		Requested days	Allocated days	From	То	
SUPERADAM		6	4	13/02/2020	17/02/2020	

Abstract:

Today, nearly every product is covered by functional coatings ranging from stability enhancers via protective coatings to colloidal sensor coatings. Ideally, such coatings are as thin as possible while maintaining their full functionality and stability. The use of latex nanoparticles (NP) in colloidal coatings is very interesting due to their easy water-processability and wide industrial application range, already in dispersive paints and adhesives. Latexes are dispersed stable colloidal polymeric particles in water phase, commonly synthesized by emulsion polymerization techniques. Block copolymer latexes fabricated via polymerization-induced self-assembly (PISA) form core-shell particles with tailored corona properties to interact with specific surfaces such as cellulose or silica. Since the properties of colloidal coatings are given by the nanostructure and morphology of the film and its surface, understanding the film formation, self-assembly and temperature stability of latex coatings systems is of great industrial and research interest.

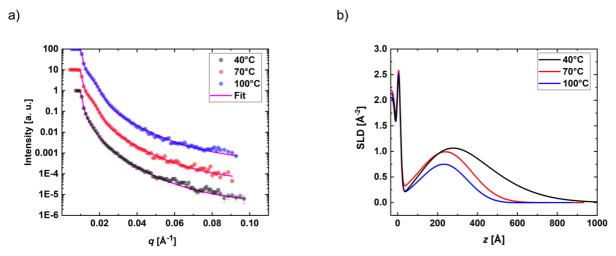
## **Experimental Report**

## Studying the percolation of soft and rigid latex nanoparticles during annealing

We used neutron reflectometry (NR) and grazing incidence small-angle neutron scattering (GISANS) at SUPERADAM to follow the density gradient and surface morphology of latex films with increasing annealing temperature (room temperature to 140°C). We used spray deposited thin films which were created at our home lab at KTH Stockholm. We used synthetized latex nanoparticles consisting of a Poly(methyl methacrylate) (PMMA) core and a 2-Dimethylaminoethyl methacrylate shell polymer. We synthetized two distinct sizes (ca. 45 nm, ca. 110 nm) of nanoparticles and prepared the samples either with protonated or deuterated core PMMA polymer. The substrate was silicon, and we deposited different nanoparticle sub-monolayers or multilayer structures. For the multilayer films we deposited a thin film of nanocellulose as porous substrate material and deposited subsequently the latex nanoparticles on top. We then studied how the nanoparticles penetrate in this porous media and how the temperature affects the nanoparticles within.

The proposed experiment as well as the proposed setup at the beamline was used without difficulties. We had two samples which did not reflect well in NR geometry due to an unforeseen bending of the silicon substrate, however enough additional samples are measured to exclude these.

We started evaluating the NR data and fitted them accordingly with the software package GenX. The sample stack is consisting of the silicon substrate, native silica layer followed by an air/polymer mixed layer and a full polymer layer. The SLDs are initially calculated from the used materials. The fitting reproduced the NR curves with high precision and the SLD is plotted versus the surface normal, see Fig. 1. This allows us the immediate access to the change in density, thickness and roughness at the different interfaces.



**Figure 1** a) NR curves for the smaller protonated PMMA core-shell latex nanoparticles on a bare silicon substrate. The fitting is performed using GenX with a multilayer fitting approach. b) Scattering length density along the surface normal for the fitted samples in a).

Layer-by-layer deposition and subsequent annealing can be considered as a simple step to functional thin films. Using NR and GISANS we are thus able to address if questions as the change in density and morphology during annealing and can correlate these phenomena to the surface functionality. The use of nanocellulose substrates on the other hand increase the use of such functional nanoparticle in future paper-based sensors, or other organic electronics. The aim of our experiment was to create multilayer thin films using different nanoparticles and substrates which was successfully performed. Further water contact angle, AFM, SEM and FTIR measurements needs to be done to fully correlate the changes in nanoparticle evolution on the different substrates. The measurements were highly successful, and the data will constitute a central part in a manuscript that we are starting to prepare now. From ILL/Uppsala University as co-author in any publication will be Alexei Vorobiev.