

Nanomanufacturing Focus Topic

Room: Hall D - Session NM-ThP

Nanomanufacturing Poster Session

NM-ThP1 Nanoscale Release Effect of Antisticking Layer for Nanoimprint Resin Characterized by Scanning Probe Microscopy. *M. Okada*, University of Hyogo, Japan, *M. Iwasa*, SII Nanotechnology Inc., *K. Nakamatsu*, *K. Kanda*, *Y. Haruyama*, *S. Matsui*, University of Hyogo, Japan
Nanoimprint lithography (NIL) has a capability to fabricate nanostructure devices with a high-throughput and a low cost. Nanoimprint lithography molds are coated with an antisticking layer so that they do not come in contact with the adhesion of replication materials. Friction and adsorption occurs between the nanostructure mold and nanoimprint resin during demolding. Measuring the nanoscale frictional force and adsorptivity between the antisticking layer and resin is therefore important. In this paper, we measured the nanoscale frictional force and adsorptivity between a cantilever and nanoimprint resin by scanning probe microscopy (SPM) using Si cantilevers with and without antisticking layer. We used a self-assembled monolayer (SAM) consisting of a silane-coupling agent with fluoropolymer (OPTOOL DSX: Daikin industries) as an antisticking layer. The thermoplastic resin (PMMA; OEER 1000: TOKYO OHKA KOGYO Co.) and the photosensitive resin (PAK-01: Toyo Gosei Co.) were also used. In the SPM measurement using Si cantilever, results were obtained between the Si and NIL resin. On the other hand, the measurement results between the antisticking layer and resin were obtained by SPM using the cantilever with an antisticking layer. The frictional force of PMMA and PAK-01 measured using the cantilever with an antisticking layer are respectively 0.7 and 0.6 times lower than those using the cantilever without the antisticking layer. The adsorptivity of PMMA and PAK-01 measured by SPM using the cantilever with an antisticking layer are 0.4 and 0.6 times lower than those using the cantilever without the antisticking layer. These results show that SPM measurement using the cantilever with and without antisticking layers can be applied to evaluate the nanoscale release effect between an antisticking layer and nanoimprint resins.

NM-ThP2 Ultrasonic Nanoimprint on Engineering Plastics. *H. Mekarū*, *M. Takahashi*, National Institute of Advanced Industrial Science and Technology (AIST), Japan

We have developed a new ultrasonic nanoimprint technology that is superior to the current thermal and UV nanoimprint technologies. In this method an ultrasonic vibration is impressed in the direction of loading force during a molding operation at room temperature. Our nanoimprint system employed a magnetostriction actuator capable of generating ultrasonic vibration with frequencies and amplitudes ranging from DC to 30 kHz, and from 0 to $\pm 30 \mu\text{m}$ respectively. Here an electroformed-Ni mold consisting of dot and line/space patterns with a minimum width of 500 nm was employed to endure the mechanical stress by the ultrasonic vibration. The mold was mounted onto the ultrasonic generator with a photoresist. We report on the results of ultrasonic nanoimprinting on various engineering plastics. At first, several optimized imprinting conditions were investigated by using polyethylene terephthalate (PET, $T_g = 75^\circ\text{C}$) with a comparatively low glass transition temperature (T_g) in engineering plastics. When the frequency of the ultrasonic vibration was varied in steps of 0.1 - 10 kHz, and the amplitude in steps of 0.5 - 3 μm , the height of the imprinted pattern was found to rise with the increase in the frequency and the amplitude. In this case, a rubber sheet serving as a buffer was interposed between the thermoplastic sheet and the bottom loading stage to keep the contact force of mold patterns on the thermoplastic uniform. By employing four kinds of rubbers the hardness of the buffer material was changed to 32, 57, 80, and 90°. When the low repulsion rubber sheet in the hardness of 57° was used without ultrasonic vibration, the height of the imprinted pattern reached a maximum. However, in the presence the ultrasonic vibration the optimized buffer material was a sheet of urethane in the hardness of 90°. Then under those same molding conditions, polycarbonates (PC, $T_g = 150^\circ\text{C}$) and polymethyl methacrylates (PMMA, $T_g = 105^\circ\text{C}$) were successfully imprinted and the pattern was observed by SEM; although in the absence of the ultrasonic vibration it was not possible to mold. The heights of the imprinted pattern were measured to be 1 μm in PET, 750 nm in PMMA, and 370 nm in PC. The assisting effect of the ultrasonic vibration was found to be inversely proportional to T_g . Therefore, it is inferred that the thermoplastic was softened locally with the frictional heat generated by the ultrasonic vibration.

NM-ThP3 Atomic Layer Etching of Cl-Adsorbed GaAs by Using a Low-Angle Forward Reflected Ne Neutral Beam. *W.S. Lim*, *J.B. Park*, *G.Y. Yeom*, Sungkyunkwan University, Korea

GaAs compound-based semiconductors are currently investigated for various devices such as high efficiency solar-cell, high mobility electronic devices, optical devices, etc. due to direct band structure, high electron mobility, high operating temperature range, etc. For the processing of gallium arsenide (GaAs), conventional dry etching processes such as chlorine-based inductively-coupled plasma (ICP) are used but they tend to cause physical damage on the surface due to the irradiation of high energy ions during the etching, and which results in the degradation of the devices. Therefore, the removal of surface damage during the processing is essential and, among the various dry etching processes, atomic layer etching (ALET) has been studied to realize atomic scale etch-rate controllability of GaAs without physically damaging the surface of GaAs. In this study, the characteristics of ALET for (100)/ (111) GaAs have been investigated as functions of Cl_2 pressure during the adsorption stage and Ne neutral beam irradiation dose during the desorption stage. By using the Ne neutral beam dose and the Cl_2 pressure higher than the critical values of 3.03×10^{16} atoms/cm²·cycle and 0.4 mTorr, respectively, one monolayer etching condition of 1.41 Å/cycle for (100) GaAs and 1.63 Å/cycle for (111) GaAs could be obtained through the adsorption of one monolayer of Cl_2 during the adsorption stage and desorption of all of the chlorides formed on the surface by the sufficient Ne neutral beam irradiation. At the monolayer etching conditions, the surface roughness was the lowest and was similar to that of as-received GaAs. Therefore, the use of ALET condition enabled us to control the etch depth with an atomic-scale precision without damaging the surface physically or chemically.

NM-ThP4 Fabrication of a Highly-Oriented Line Structure on an Aluminum Surface and the Nanoscale Patterning on the Nanoscale Structure using Highly-Functional Molecules. *Y. Watanabe*, *H. Kato*, *S. Takemura*, *H. Watanabe*, *K. Hayakawa*, *S. Kimura*, *D. Okumura*, *T. Sugiyama*, *T. Hiramatsu*, *N. Nanba*, *O. Nishikawa*, Kanto Gakuin University, Japan, *M. Taniguchi*, Kanazawa Institute of Technology, Japan

The surface of an Al plate was treated with a combination of chemical and electrochemical processes. Chemical treatment with acetone and successive electrochemical process were performed on the aluminum surface. Dynamic force microscopy (DFM) measurements after the chemical treatment demonstrated that a fibril-like structure with random widths in the same orientation was initially formed. The successive electrochemical process made the initial fiber-like structure more ordered and finer. The anodization successfully created a nanoscale highly-oriented line structure on an Al surface. The distance between the oriented lines was estimated as 30-40 nm. Furthermore, the present work intended to make an organic-inorganic nanoscale pattern using this nanoscale structure. Copper phthalocyanine (CuPc), fullerene C60, and polyaniline were selected in fabrication of functional nanoscale patterning. CuPc and C60 molecules were deposited on the highly-oriented line-structure on an aluminum surface. A toluene droplet containing CuPc molecules was cast on the Al plate and extended on the surface. CuPc deposition on the Al surface was made by evaporation of toluene. Cross section analysis of the DFM measurements clarified that each row line was filled with CuPc molecules because the line structure was clearly observed after the deposition. The depth of the row line became shallow and in some area the row width became narrower after the CuPc deposition. X-ray photoemission spectroscopy (XPS) measurements clarified that N 1s and Cu 2p lines appeared, which also supports the CuPc deposition on the nanoscale structure creating a nanoscale organic line pattern. The spectral profile difference of Al 2p pre- and after CuPc deposition suggested the molecular-surface interaction. Furthermore, the fabrication of a polyaniline nanowire along the row channel of the highly-oriented line structure was performed. A droplet of HCl solution containing aniline molecules was cast and extended on the nano-structured Al plate. Successively, a droplet containing APS was put and extended on the surface. DFM and XPS clarified that aniline molecules were polymerized in the channel. X-ray photoemission spectroscopy (XPS) and Fourier transform spectroscopy measurements supported the polymerization of the nanowire. This work was supported by High-Tech Research Center Project aided by MEXT.

NM-ThP5 Fabrication and Photoluminescent Properties of Nanostructured Al Surfaces Topped by Polythiophene Film and Copper Phthalocyanine. *H. Kato, S. Takemura, A. Ishii, Y. Takarai, H. Koba, Y. Watanabe, T. Hiramatsu, N. Nanba,* Kanto Gakuin University, Japan

Nanostructures such as linked-crater structure and highly-oriented line structure were fabricated on an Al surface by combination process of chemical and electrochemical treatments. Conducting polymer polythiophene nanofilm growth on the nanostructured Al surface was conducted by an electrochemical synthetic method. Copper phthalocyanine (CuPc) doping in the nanofilm was conducted by a diffusion method. Photoluminescent properties of the nanostructured Al surfaces and the surfaces topped by the nanofilm and CuPc were investigated. A nanoscale linked crater structure was fabricated on an Al surface by treatment with Semi Clean in order to create crater-shaped erosion on the surface as an initial structure. The successive electrochemical anodization in H₂SO₄ solution created a nanoscale finer linked-crater structure on the surface. The crater size was estimated as 80-150 nm in diameter. Dynamic force microscopy (DFM) measurements clarified that small pores with 9 nm in diameter were created in each crater. Regarding the highly-oriented line structure, the anodization process applied to the original fiber-like surface structure on the Al plate. The anodization fabricated the finer line structure on the Al surface. The line distance was estimated as 40 nm. In the case of polythiophene film growth on the linked-crater structure, nanofilm was grown along the crater structure and the secondary structure remained demonstrated by DFM. Photoluminescence measurements on the linked-crater structure showed that five emission peaks appeared while no emission peaks were observed in the case of the original Al surface. Photoluminescence measurements on the polythiophene nanofilm on the linked-crater structure showed that the five emission peaks clearly appeared. The emission wavelengths were shifted. Furthermore, the photoluminescence measurement on the nanofilm doped with CuPc showed that the five emission peaks were enhanced. The photoluminescence measurements clarified that the nanostructured Al surface and the surface topped by nanofilm and CuPc molecules showed the characteristic emission properties for creation of functional hybrid materials. This work was supported by High-Tech Research Center Project aided by MEXT.

NM-ThP6 Synthesis of Various Shaped Pt and FePt Nanoparticles. *N. Shukla, M.M. Nigra, M. Bartel, T. Nuhfer, A.J. Gellman,* Carnegie Mellon University

We report the synthesis and self-assembly of controlled shapes and sizes of Pt nanoparticles using a high pressure synthesis technique. Our work suggests that solvent plays an important role in tailoring the shapes of Pt nanoparticles. Solvents can impact the synthesis because their boiling points dictate reaction trend and they also play a role as surfactants. Using various types of solvents under high pressure we have been able to synthesize Pt spheres, nanocubes and hexagons. 3D shapes of these nanoparticles are characterized using angle resolved high resolution TEM imaging. In addition, we report one step synthesis of core shell structures of cubic Pt nanoparticles. In this synthesis cubic Pt nanoparticles are the core and hexagonally shaped iron oxide/Fe is the shell. The most interesting part of this synthesis is that the shape of the core particles and shell is different. In most cases the core shells are either spherical or they acquire the shape of the nanoparticles. In this work we have achieved formation of cubic Pt nanoparticles with well hexagonal shaped iron oxide. These types of multiphase nanoparticles can have interesting applications in catalysis

NM-ThP7 Analytical Model for Geometrical Characteristics Control of Laser Sintered Surfaces. *Y. Ioannou, K. Polychronopoulou, C.C. Doumanidis,* University of Cyprus

Selective laser sintering (SLS) is an additive rapid manufacturing technique where high-power laser is used to fuse small particles into a specified 3-dimensional geometry. The goal of this work was to develop an analytical model for the SLS manufacturing processes, in order to control the geometrical characteristics of the sintered areas when iron/copper (Fe/Cu) powder alloy is used on a flat substrate. Powder particles are subject to melting by the laser energy and form a liquid globule, which solidifies as the laser beam spot moves on the substrate. The model is based on a system of lumped energy and mass balances, and the temperature field is computed via Green's function formulations by convolution of impulsive heat inputs at specific times and locations. The Green's function is dependent on the material conductive properties, substrate geometry, and boundary conditions. In considering the generated surface tensions due to powder melting, an approximation of the sintered area geometry after solidification of the material is performed via fluid dynamic equilibrium theory. Independent process parameters considered to govern the intensity of energy delivered to the powder material include laser power, beam spot size, beam velocity, hatch spacing and scan line length. Computational

simulations are calibrated via and validated against experimentally fabricated SLS samples with simple deposition geometries.

NM-ThP8 Low Damage Etching of Ge₂Sb₂Te₅ by using Halogen-Based Neutral Beams. *S.-K. Kang,* Sungkyun Advanced Institute of Nano Technology (SAINT), Korea, *B.J. Park, S.W. Kim, T.H. Min,* Sungkyunkwan University, Korea, *G.Y. Yeom,* Sungkyun Advanced Institute of Nano Technology (SAINT), Korea

Phase-change random access memory (PRAM) has made a prominent progress in memory performance and has brought a bright prospect for the next generation nonvolatile memory technologies due to its excellent advantages. One of the chalcogenide-based phase-change materials that have been paid attention for nanoscale nonvolatile memories is Ge₂Sb₂Te₅ (GST) which has advantageous properties such as high-speed phase transformation and high degree of read-write cyclability without any compositional change between different phases. Ge-Sb-Te based materials are known to be form halogenations easily and apt to be damaged when exposed to plasma in the etch process. In this work, we present X-ray photoelectron spectroscopy (XPS) data obtained from the surface of blank GST and etched GST features after etching in the halogen inductively coupled plasmas (ICP) such as CF₄, Cl₂ and HBr to investigate the extent of halogenation damage. And we also investigated surface damage of the GST etched by halogen-based neutral beam to compare with ICP process. Recently, the neutral beam etching (NBE) method is used by several researchers to avoid the charge-related damage. The damage related to charging can be eliminated because, compared to the conventional reactive ion etching, no ions participate in the etch process. Our neutral beam was formed using a low angle forward reflection of a parallel ion beam obtained by an ion gun. The XPS compositional depth profiling for the blank GST and the topological XPS analysis for the patterned GST were investigated after etching in the ICP and the NBE system to prove less damage process condition. In the etching of GST in halogen-based ICP and NBE system, the spectra of Ge, Sb and Te are shifted to higher binding energy region with different extents for different halogen-based plasmas. It indicates that GST film received different extent of damage for the halogen-based plasmas. Significantly, we could observe the less changes of GST-degradation when the NBE was applied to etch the GST.

NM-ThP9 Templated Self-Assembling Conducting Polymers as a Substrate for Nanoscale Design. *M.V. Lee, J.P. Hill, K. Ariga,* National Institute for Materials Science, Japan

Various strategies are being utilized to revolutionize nanoscale fabrication. A method based on a series of novel conductive polymers is demonstrated. Several functional groups can be incorporated into structures produced to affect both self-assembly and properties of the resulting polymer structures. Three dimensional structures for sensing and biological applications are envisioned.

NM-ThP10 Plasma Treatment of PE Powder - From Laboratory Experiments to Production Plant. *P. Spatenka, J. Hladik,* Technical University of Liberec, Czech Republic, *J. Pichal,* Czech Technical University of Prague

Plasma modification of powder has recently attracted much interest because of new perspectives of the interfacial properties supervision. Plasma modified powder was used as a filler for composite materials or for production of components using roto-molding technique. Parts sintered from the plasma modified polyethylene powder preserved high surface tension, which allowed e.g. direct painting or adhesive bonding without any additional pretreatment. Plasma modification also significantly enhanced adhesion of the polymer to the substrate. Various methods for powder treatment have been reported for low-pressure plasma treatment of powder including fluidized bed or reactors with mechanical stirring. Based on laboratory experiments and analysis of various treatment methods we proposed a concept for industrial scale production of plasma-modified powder. The principle of the industrial-scale reactor will be presented. Production capacity and cost estimation will be also presented. Partial support of the projects KAN101120701 and MSM 4674788501 and GACR 106/08/1665 is greatly acknowledged.

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