Electrodeposition of inorganic/organic hybrid thin films from one pot containing all the chemical ingredients, what we named “Electrochemical Self-Assembly” (ESA) is a good platform to test “chemistry” between the inorganic and organic constituents. When organic molecules added to the electrolytic baths for electrodeposition of inorganic compound thin films find stable adsorption to the inorganic surface, their hybrid thin films are obtained and morphology as well as crystal orientation of the inorganic crystals are significantly altered as a consequence of adsorption of organic molecules onto specific crystal facets. Such hybrid thin films are certainly interesting as new materials, since concerted functionalities are anticipated from intimate interaction between inorganic and organic constituents.

The leading example is the ESA of nanostructured ZnO/dye hybrid thin films. When water soluble organic dye molecules bearing hard Lewis basic anchors such as carboxylate, sulfonate and phosphonate are added to the bath for cathodic electrodeposition of ZnO (aqueous solution of Zn(NO₃)₂ or ZnCl₂ saturated with O₂), the anchors coordinate to hard Lewis acidic sites of Zn(II) of ZnO, so that colored hybrid thin films are spontaneously formed. Especailly suited was carboxylated xanthene dye such as Eosin Y (EY), because of the ordered strong dipole moment of the cationic DAS. Nucleophilicity of EY was further enhanced by introducing an extra carboxylic acid group (carboxy-Eosin Y, CEY) and also when it was electrochemically reduced, so that loading of electrochemically reduced CEY became purely diffusion-limited, namely, 100% of CEY that reached the electrode surface “sank” into the film. It is thus obvious that these dyes are not passively occluded into ZnO during its electrochemical growth, but actively participa in the electrochemical Self-Assembly (ESA) is a good platform to test “chemistry” between the inorganic and organic constituents.

The primary condition of ESA is the “good chemistry” between inorganic and organic constituents to precipitate them together, although the degree and the mode of chemical affinity can alter the mechanism and thereby the product hybrid thin films. For example, the difference of adsorption stability could change the probability of dye loading, namely, the portion of precipitated dye with respect to the diffusion flux. Such factors affecting the ESA can be analyzed and quantified to elucidate the mechanism and yield “tuning knobs” to achieve an entire control over the process of ESA. It is not an easy task but one has to reach a full understanding of the mechanism to sophisticate ESA as an industrially scalable and controllable process.

As a counterpart of the ZnO-based ESA, electrodeposition of CuSCN also provides a platform to test its ESA with organic dyes. While ZnO is typically a n-type semiconductor, CuSCN is a wide bandgap p-type semiconductor, successfully applied as a hole conducting layer in thin film solar cells. Once again, the right chemistry is needed for the dyes to be used for successful ESA. Fluorescein isothiocyanate (FLNSC) with a soft Lewis basic –NCS was the right one among the xanthene dyes to coordinate to the soft Lewis basic Cu(I) site of CuSCN, while carboxylated xanthene dyes such as EY completely failed to yield hybrid thin films with CuSCN. Hard and soft acid and base (HSAB) principle is thus one of the keys to explain the difference for the ESA between ZnO and CuSCN systems. Another mechanism of ESA of CuSCN/dye hybrid is the substitution of Cu⁺ ions with cationic dyes. Indeed, many of cationic dyes achieved successful ESA with CuSCN. Especially interesting among them was the ESA with an addition of 4-(N,N-dimethylamino)-4′-(N′-methyl)stilbazolium tosylate (DAST). DAST crystal is known for its non-linear optical (NLO) property as a THz emitter because of the ordered strong dipole moment of the cationic DAS. While all the other ZnO/dye and CuSCN/dye systems in which fluorescence...
Experimental

Copper(II) perchlorate hexahydrate ([Cu(ClO$_4$)$_2$]•6H$_2$O, 98%, Aldrich), lithium thiocyanate dihydrate (LiSCN•2H$_2$O, 95%, Kishida), lithium perchlorate (LiClO$_4$, 98%, Wako) and methanol (99.8%, Wako) were used as purchased. Crystalline powder of 4-(N, N-dimethylamino)-4′-(N′-methyl) stilbazolium tosylate (DAST) was synthesized according to the procedure published elsewhere.$^{21,22}$

Electrolytic bath for the electrodeposition of CuSCN was prepared to vary the concentration of the active species, [CuSCN]$^{+}$, between 0.25 and 5.0 mmol dm$^{-3}$ by dissolving CuCl$_2$.6H$_2$O and LiSCN in a 1: 1 ratio to methanol, in the concentration range of 0.25 – 5.0 mmol dm$^{-3}$, which also contained 0.1 mol dm$^{-3}$ LiClO$_4$ as the supporting electrolyte. On the other hand, the concentration of DAST to be added to the bath was independently controlled as 10, 20, 30, 40, 50, 70 and 100 μmol dm$^{-3}$. Fluorine-doped SnO$_2$ (FTO) coated conductive glass substrate (Asahi-DU 10 Ω/sq.) cut into 20 × 35 mm was ultrasonically cleaned sequentially in Decon detergent, acetone, and 2-propanol to be stored. It was activated in a 45% HNO$_3$ for 2 min and rinsed with distilled water before it was mounted to a home-made RDE setup via a specially designed attachment (as shown in Fig. 3). Electrical contact between the FTO surface and the attachment was made by putting two strips of Cu tape and inserting a small amount of In-Ga eutectic. The active area of the FTO glass RDE as the working electrode was regulated to 1.54 cm$^2$ by a masking tape (Nitto Denko N-380) with a 14 mm diameter round hole concentric to the rotating center (Fig. 3b). While the FTO mounted attachment is screwed to the rotating shaft of the motor drive, the lid holding all the electrodes and gas bubbler is assembled with double-walled flask, for which the inner flask contains the sample solution whereas the outer serves as a water jacket for temperature control (298 K). A Pt wire and an Ag/AgCl electrode served as counter and reference electrodes, respectively. Potentiostatic cathodic electrolysis at +0.2 V (vs. Ag/AgCl) with rotation speeds varied as ω = 100, 300, 500, 700 and 900 rpm, for 300 s and under air yielded CuSCN/DAS hybrid thin films with variation of CuSCN to be electrodeposited and DAS to be loaded. An electrochemical system (Hokuto Denko HSV-110) was used for potential control and current monitoring.

The amount of DAS loaded into the hybrid thin films was determined by dissolving the entire film (1.54 cm$^2$ projected area) into 10 ml of 7 mol dm$^{-3}$ ammonia and measuring its absorption spectrum on a SHIMADZU Solid Spec-3700 spectrophotometer for the peak of DAS complex ($\lambda = 455$ nm, $\varepsilon = 2.80 \times 10^3$ M$^{-1}$ cm$^{-1}$ in ammonia, as determined separately). The amount of CuSCN precipitated as a thin film has also been estimated spectroscopically according to Lambert-Beer’s Law by measuring the absorbance for the d-d transition of $[\text{Cu(NH}_3)_2\text{Cl}]^+$ complex ($\lambda = 638$ nm, $\varepsilon = 137$ M$^{-1}$ cm$^{-1}$ as determined by dissolving CuCl$_2$.6H$_2$O in 7 mol dm$^{-3}$ ammonia) spontaneously formed after film dissolution.

In order to check the location of DAS, namely, DAS either entrapped in CuSCN or phase separated from CuSCN, the electrodeposited hybrid thin films were soaked in N,N-dimethylacetamide (DMA) at room temperature for overnight. The degree of DAS extraction has been checked by measuring transmission absorption spectra as well as observing the surface morphology on a HITACHI SU8000 field emission scanning electron microscope (FE-SEM), before and after the DMA treatment.

Results and Discussion

Previous study has found the transition from diffusion limited to surface reaction limited loading of DAS around the DAST concentration of 60 μmol dm$^{-3}$ in the bath containing the active species for electrodeposition of CuSCN, namely, the [CuSCN]$^{+}$ complex, at 2.5 mmol dm$^{-3}$. Since electrodeposition of CuSCN described by...
Figure 3. The setup for electrodeposition of CuSCN/DAS hybrid thin films used in this study; a sketch of the electrode attachment with a rectangular 1.8 mm deep recess to which an FTO glass substrate is inserted to achieve a flush surface (a), a sketch to show how the electrical contact and regulation of active surface area are made by use of conductive Cu and insulating masking tapes, respectively (b), and the picture of the assembled RDE system in which the electrode attachment mounting an FTO glass is screwed to the motor drive shaft, Pt wire and Ag/AgCl are placed as working, counter and reference electrodes, respectively, while inner and outer flasks serve as the cell to contain sample solutions and water jacket for temperature control, respectively (c). This system allows thin film electrodeposition with high homogeneity and reproducibility under controlled mass transport, atmosphere and temperature, as well as use of various substrates including optically transparent and back side insulating FTO glass used in this study to achieve direct optical characterization and use of the product thin films.
Eq. 1 proceeds as limited by diffusion of [Cu(SCN)]⁺ with a Faradic efficiency of practically 100%. The rate of CuSCN formation can be tuned by changing the concentration of [Cu(SCN)]⁺ in the bath and by rotation speed of the RDE, irrespective of the DAST concentration in the bath, although DAST addition in extreme high concentrations may result in a slight decrease of current as well as the Faradic efficiency. The current density, \( J \) (A cm⁻²), at the RDE during the electrolysis can therefore be simply expressed by Levich equation as,

\[
J = 0.62 \times F \times C_{\text{comp}} \times D_{\text{comp}}^{1/2} \times \nu^{1/2} \times \omega^{1/4}
\]

where, \( F \) is the Faraday constant (96,485 C mol⁻¹), \( C_{\text{comp}} \) and \( D_{\text{comp}} \) are the bulk concentration (mol cm⁻³) and diffusion coefficient (2.26 × 10⁻⁶ cm² s⁻¹) in methanol at 298 K [22] of [Cu(SCN)]⁺ precursor complex, respectively, \( \nu \) is the kinetic viscosity of the medium (0.687 × 10⁻⁴ cm² s⁻¹ for methanol at 298 K [28]) and \( \omega \) is the angular speed of rotation (rad s⁻¹). Thus, \( J \) is directly proportional to \( C_{\text{comp}} \) and to the square root of \( \omega \). Assuming the constant Faradic efficiency of 100% for the electrodeposition of CuSCN, its rate of precipitation \( (P_{\text{CuSCN}} \text{ mol cm}^{-2} \text{ s}^{-1}) \) is simply described as,

\[
P_{\text{CuSCN}} = \frac{J}{F} = 0.62 \times C_{\text{comp}} \times D_{\text{comp}}^{1/2} \times \nu^{1/2} \times \omega^{1/4}
\]

for which the diffusion coefficient of DAS, \( D_{\text{DAS}} \), has previously been determined as 1.25 × 10⁻⁶ cm² s⁻¹ in methanol at 298 K. [20]

With the above mentioned ideas in mind, several hybrid thin films were electrodeposited on variation of \( C_{\text{comp}} \) (while keeping \( \omega \) = 500 rpm) and \( \omega \) (while keeping \( C_{\text{comp}} \) = 2.5 mmol dm⁻³), with a constant \( C_{\text{DAS}} = 10 \mu\text{mol dm}^{-3} \), well below the previously found border, thus to represent the conditions for diffusion-limited loading of DAS. The amount of both CuSCN and DAS electrodeposited during the electrolysis for 300 s (thus are equal to \( 300 \times P_{\text{CuSCN}} \) and \( 300 \times P_{\text{DAS}} \), respectively) are plotted against \( C_{\text{comp}} \) and \( \omega^{1/2} \) (Fig. 4a), for which the horizontal scaling is aligned to yield the same diffusion flux density of [Cu(SCN)]⁺, in order to facilitate checking their giving the same influence to \( J \) according to Eq. 2. It is obvious that all the plots for \( P_{\text{CuSCN}} \) fall onto one straight line, since \( J \) is simply proportional to the flux density, which can be tuned equally either by changing \( C_{\text{comp}} \) or \( \omega^{1/2} \). On the other hand, a totally different dependence has been found for \( P_{\text{DAS}} \). The loading of DAS is proportional to \( \omega^{1/2} \) as expected from Eq. 4, but the change of \( C_{\text{comp}} \) gives no influence to result in a constant \( P_{\text{DAS}} \). The same analysis has been carried out for the hybrid thin films electrodeposited with \( C_{\text{DAS}} = 100 \mu\text{mol dm}^{-3} \), well above the border to represent the surface reaction-limited regime. The plots of \( P_{\text{CuSCN}} \) and \( P_{\text{DAS}} \) against \( C_{\text{comp}} \) and \( \omega^{1/2} \) are shown in Fig. 4b. Once again, the linear proportionality is established for \( P_{\text{CuSCN}} \) against both \( C_{\text{comp}} \) and \( \omega^{1/2} \), as reasonably expected from Eq. 3. However, \( P_{\text{DAS}} \) now also appears linearly proportional to both \( C_{\text{comp}} \) and \( \omega^{1/2} \), along with the increase of CuSCN precipitation rate. When DAS loading is limited by the formation of surface complex between CuSCN and DAS, \( P_{\text{DAS}} \) is expected to be proportional not only to \( C_{\text{DAS}} \) (whose surface concentration to be equal to that in the bulk) but also to \( P_{\text{CuSCN}} \), as the newly formed CuSCN provide sites for coordination of DAS. The switching from the diffusion-limited to the surface reaction-limited regimes was nicely observed when a moderate \( C_{\text{DAS}} = 30 \mu\text{mol dm}^{-3} \) was chosen (Fig. 4c). \( P_{\text{DAS}} \) linearly increases along the increase of \( C_{\text{comp}} \) up to 2 mmol dm⁻³ and then levels off to become constant. When \( C_{\text{comp}} \) is kept low, \( C_{\text{DAS}} = 30 \mu\text{mol dm}^{-3} \) is above the threshold to fall into the surface reaction-limited regime, but is small relative to \( C_{\text{comp}} \) when it is above 2 mmol dm⁻³, so that the diffusion-limited loading operates. These results already indicate that the switching is dependent on the relative concentration of \( C_{\text{DAS}}/C_{\text{comp}} \).

In order to confirm the switching of dye loading mechanism, the product thin films were analyzed to clarify the location of DAS in

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**Figure 4.** Amount of the electrodeposited CuSCN (300 × \( P_{\text{CuSCN}} \)) and DAS (300 × \( P_{\text{DAS}} \)) plotted against \( C_{\text{comp}} \) (while fixing \( \omega = 500 \text{ rpm} \)) and \( \omega^{1/2} \) (while fixing \( C_{\text{comp}} = 2.5 \text{ mmol dm}^{-3} \)) for \( C_{\text{DAS}} = 10 \) (a), 100 (b) and 30 \( \mu\text{mol dm}^{-3} \) (c). The horizontal scalings of \( C_{\text{comp}} \) and \( \omega^{1/2} \) are aligned for the same flux density of [Cu(SCN)]⁺, so that the same \( J \) and \( P_{\text{CuSCN}} \) are expected from Eqs. 2 and 3, respectively. Thus, the linear fitting for \( P_{\text{CuSCN}} \) is made by taking all the plots into account, for both the series to vary \( C_{\text{comp}} \) and \( \omega^{1/2} \). The same is true for \( P_{\text{DAS}} \) in (b), but the fitting was made independently for the series to vary \( C_{\text{comp}} \) and \( \omega^{1/2} \) for those in (a) and (c). The plots in (c) for variation of \( C_{\text{comp}} \) were divided in two segments, those between 0.5 and 2.0 mmol dm⁻³ and those between 2.0 and 4.0 mmol dm⁻³ for the fitting, as the switching of the loading mechanism occurs at around 2.0 mmol dm⁻³.
CuSCN (entrainment vs. segregation) for those electrodeposited with a moderate $C_{\text{DAS}} = 40 \, \mu \text{mol dm}^{-3}$ on gradually increasing $C_{\text{comp}}$. Fig. 5 shows SEM pictures of the thin films before and after the DMA treatment, while Fig. 6 shows the absorption spectra. With low $C_{\text{comp}}$ of 1.0 mmol dm$^{-3}$, rectangular shape of the deposit is clearly recognized, which we previously named as “haircomb” shape, although the teeth of the haircomb grown along the $c$-axis of $\beta$-CuSCN are not clear for this sample. When it was soaked in DMA, DAS was almost completely extracted as monitored by the significant decrease of the peak absorbance. At the same time, the film became porous due to removal of DAS. On the contrary, the thin films grown with high $C_{\text{comp}}$ (4.0 and 5.0 mmol dm$^{-3}$) are made of rugged particles which resemble with those of the pure CuSCN electrodeposited without DAS. Diffusion-limit of DAS loading was reached for these thin films as they show almost the same absorbance in the spectra. Only a small fraction of DAS could be extracted when these films were soaked in DMA. The morphology of these films also do not change much by the DMA treatment, although partial removal of DAS make their surface appear a bit sharp. It is therefore clear that the diffusion-limited loading of DAS results in its entrainment in the CuSCN grain, whereas the low $C_{\text{comp}}$ relative to $C_{\text{DAS}}$ reduces the probability of dye loading as limited by the formation of surface complex, which in turn allow re-organization of CuSCN and (DAS)(SCN) aggregate, resulting in their phase-separated unique hybrid nanostructure. This switching of the dye loading mechanism, associated with the change of the hybrid structure, does not occur abruptly but gradually, as one can find an intermediate zone. When $C_{\text{comp}}$ was raised as 2.0 and 3.0 mol dm$^{-3}$, the transition from the rectangular particle to the rugged particle is nicely observed. The proportion of extractable DAS gradually decreases as the loading approaches the diffusion-limit. DMA treatment did change the morphology of these films due to partial removal of DAS present as phase separated aggregates.

Having confirmed the different dependence of $P_{\text{DAS}}$ on $C_{\text{comp}}$ in diffusion-limited (independent) and surface reaction-limited (dependent) regimes, the rate of DAS loading ($P_{\text{DAS}}$) is plotted against $C_{\text{comp}}$ for variation of $C_{\text{DAS}}$ as shown in Fig. 7. Two distinctively different regions are recognized, where $P_{\text{DAS}}$ linearly increases on increasing $C_{\text{comp}}$, and where it stays constant irrespective of $C_{\text{comp}}$. The transition between these two regions does not appear sharp but is rounded, so that linear fits have been applied to the regions with good linearity (indicated by solid lines), extrapolated (indicated by dashed line) to determine the border from the intersection. The border for the transition moves toward higher $C_{\text{comp}}$ as $C_{\text{DAS}}$ gets higher. With the highest $C_{\text{comp}}$ values for the switching transition from $C_{\text{comp}}$ as 1.0, 2.0, 3.0, 4.0 and 5.0 mmol dm$^{-3}$, while fixing $C_{\text{DAS}} = 40 \, \mu \text{mol dm}^{-3}$.
should also be noticed that the switching can also be calculated from the critical molar ratio of DAS/CuSCN of the product hybrid thin film for the loading mechanism achieved in this study already greatly contributes to the design of hybrid materials.

Limit of DAS loading for the CuSCN/DAS system, which should then provide useful information to verify the above-mentioned hypothesis. The model to explain the dye loading mechanism developed in this study should be further verified by collecting data with detailed variation of the parameters. Diffusion is thermally activated so that temperature dependence of the switching border also needs to be checked. Establishing a model to analyze the surface reaction is an ongoing challenge for the full understanding. The complex formation is either kinetically limited by bond formation of DAS to CuSCN surface, or by the stability of coordination if the surface concentration of DAS is always equilibrated. Having succeeded with such sophistications, it becomes very interesting to examine the applicability of the model to various CuSCN/dye systems taking dyes else than (but similar to) DAS, with higher or lower affinity to CuSCN. Concerted new functionalities and use of the materials for device applications, in connection to hybrid nanostructure and chemical composition, are of course also in our interest. As stated earlier, the phase-separated CuSCN/DAS hybrid thin films produced under the surface reaction-limited regime exhibited more efficient concerted photoluminescence than those grown under the diffusion-limited regime resulting in the occluded DAS. Thus, the understanding of the switching of the dye loading mechanism achieved in this study already greatly contributes to selective design and synthesis for high functionalities. For the rapid technological development, it is very important to obtain the whole picture of the mechanism of ESA, so that we can tune the film synthesis as we need to maximize the device performance.

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