

STUDY OF THE BINDER DISTRIBUTION IN AN ELECTRODE DURING DRYING

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1. Introduction

Batteries are currently one of the more important parts in vehicles designed to provide low fuel consumption. To improve cost effectiveness, research studies on the manufacturing process are needed. Battery electrodes are manufactured from an aqueous dispersion of carbon particles, viscosity modifier and latex binder. During drying, the distribution of the binder and carbon can become non-uniform with a greater concentration of binder at the surface. It is known that this heterogeneity leads to lack of the adhesion between the electrode and the substrate. However, the distribution of components during drying has not been studied systematically and countermeasures to the problem are not known. In this study, we establish a method to capture the concentration of binder in the electrode during drying.

2. Experimental

2.1 Coating and drying

Aqueous dispersions including carbon particles (graphite), viscosity modifier (carboxymethylcellulose; CMC) and latex binder (styrene-butadiene rubber; SBR) were coated on copper foils at room temperature. The dispersion was prepared in water with a weight ratio of the solid components of 100 : 1 : 30 and a total solids loading of 45 wt%. Coatings of ~150 μm

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thickness were prepared by heated air flow in an oven at 150 °C. The surface temperature and weight were measured by a Radius thermometer and a balance integrated into the drying oven, respectively.

2.2 Specimen preparation and analysis

Figure 1 shows the method used to explore the binder distribution in the coatings during drying. A sample was taken out from the oven at a particular drying time and then quickly immersed into liquid nitrogen to lock in the distribution of components in the coating at that time. The sample was freeze dried by placing it in a chamber at low temperature (-15 °C) and vacuum (under 80 kPa) for 10 hours.

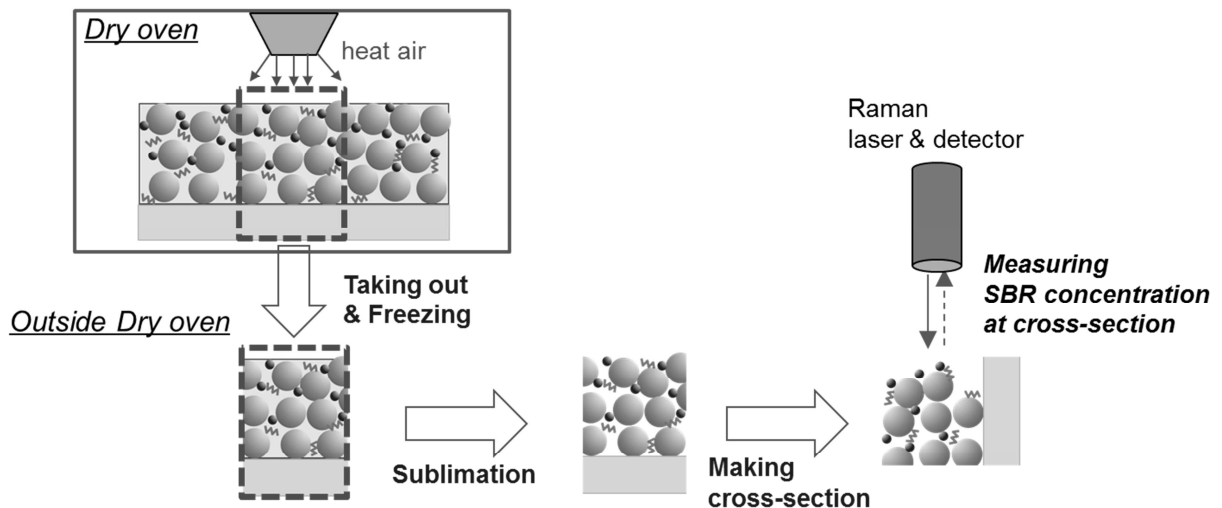


Fig. 1 Setup for the measurement of SBR profiles during drying process

A cross-section of this freeze-dried sample was then made and examined in a Confocal Raman Microscope (Alpha300R, WITec) at room temperature. Raman spectra were gathered at different positions along the cross-section. Each Raman spectrum was the average value of measured 400 spots. The SBR concentration was determined using a calibration curve in a method similar to those used to study the binder concentration at the surface of paper coatings [1,2]. The calibration curve was generated from mixtures of known quantities of SBR and graphite. A Raman peak intensity ratio of 1000 cm^{-1} (SBR)/ 1350 cm^{-1} (graphite) was used.

Scanning Electron Microscope (S-4700, Hitachi) was also used to explore the morphology of the dried samples. Cross-sections were prepared by fracturing the dried specimens under liquid nitrogen and coating with a thin layer of platinum to prevent charging.

3. Results and Discussion

Figure 2 shows the change in the surface temperature and weight of the sample recorded in situ. During the initial phase of drying, the weight loss occurs at roughly a constant rate and the temperature remains steady. After about 40 s, the temperature increases quickly to the oven temperature (150°C) and the weight loss slows considerably.

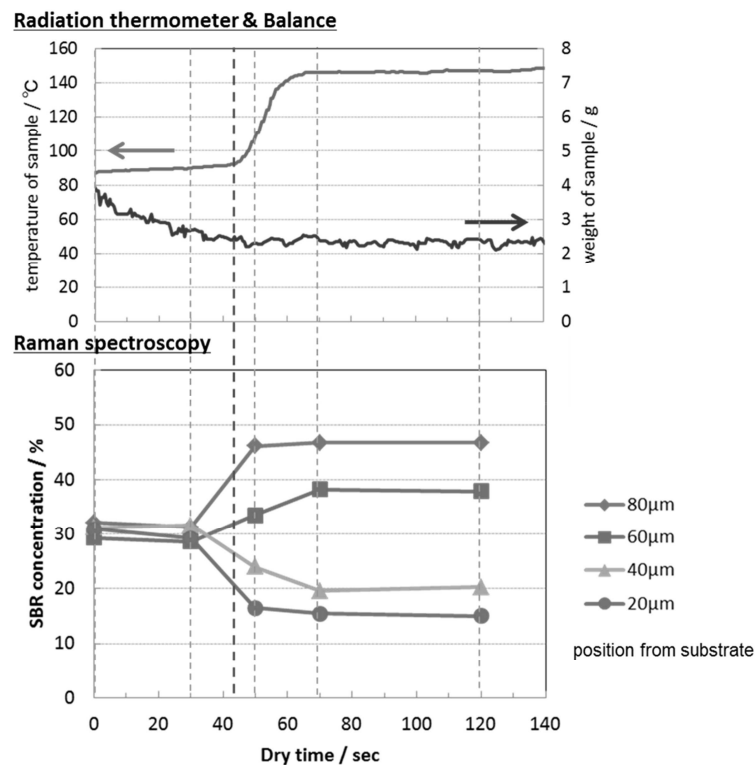


Fig.2 Measured temperature of surface and weight of sample during drying (top), and SBR concentration in layer at different drying times and positions in the coating (bottom).

Also shown in Figure 2 are the results from Raman spectroscopy of the freeze-dried samples. In the initial drying period, the SBR concentration in electrode was uniform, at the bulk concentration used to prepare the coating dispersion. However, between 30 s and 70 s there was

a dramatic change. In this later drying stager, there was more SBR near the top of the coating as compared to the bottom. The nonuniformity developed with time, but then was stable with little difference between the sample dried for 70 s as compared to that dried at 120 s. The increase in the concentration of SBR near the surface occurred later in the drying, likely due to capillary effects.

SEM images taken of the final dried coatings also indicate the SBR migration (See Figure 3). Near the surface, the microstructure consists of the large graphite particles with polymer at the particle-particle interfaces and filling some of the voids between particles. However, near the bottom of the electrode, close to the substrate, there is considerably more porosity and apparently less polymer, as judged by the lower porosity and increased angularity of the graphite particles.

More research is underway to investigate the relation between the term changing SBR concentration and the process condition, such as atmosphere temperature, pressure in the oven and the direction air flow.

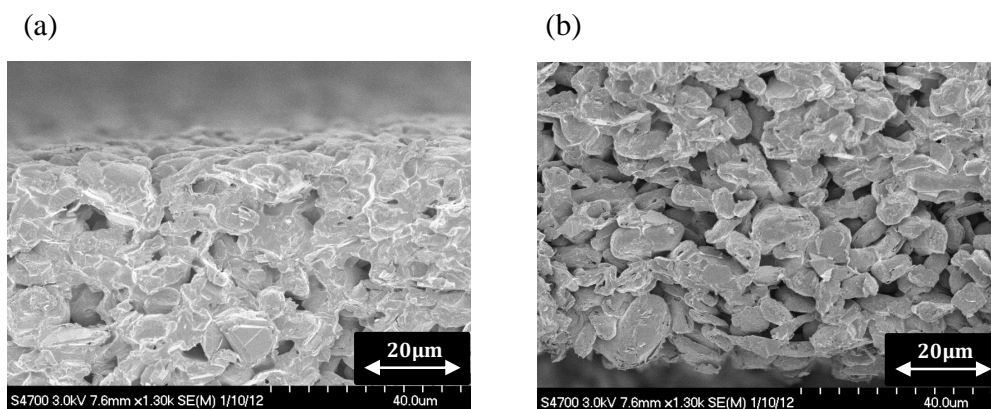


Fig. 3 SEM images of the cross-section of a dried sample: (a) near the surface, (b) near the bottom of the electrode.

References

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