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Development of Automatic TEM/SEM Specimen Preparation Instrument for Nanomaterial Dispersed in Liquid

Satoru Akai[1*], Kazuhiro Kumagai[2], Nobuo Handa[1], Akira Kurokawa[2], Yoshikazu Sasaki[1], Naoki Kikuchi[1], Shin-ichi Kitamura[1] and Hironobu Manabe[1]

[1.] JEOL Ltd., 3-1-2 Musashino, Akishima, Tokyo, Japan.
[2.] National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan.
* Corresponding author: saakai@jeol.co.jp

In recent years, regulation of nanomaterials has become active due to concern about human risk such as carcinogenicity of nanomaterial contained in industrial products. As regulations require submission of particle size distribution data of the target sample, it is required to establish a method for accurately measuring the particle size distribution of nanomaterials.

In conventional sample preparation for electron microscopy, nanoparticles dispersion liquid was dropped on a substrate with a micropipette, etc and was dried naturally. The disadvantage of this method is that agglomeration of nanoparticles called "coffee ring effect" is likely to occur and becomes obstacles to appropriate observation. As a solution to this problem, we have studied sample preparation method applying freeze-drying method. [1] We developed a new preparation instrument and report the result.

We fabricated an automatic sample preparation instrument for liquid dispersed nanoparticles based on the freeze-drying method (Fig. 1). It is equipped with an inkjet head, and it is possible to drop small droplets (1 nL or less) of dispersed nanoparticles in liquid. This instrument has a drop function which controls the dropping position by the CMOS camera and the XYZ manipulator, and it is possible to drop the dispersion liquid to an arbitrary position on the substrate. The substrate is placed in a humidity-controlled vacuum chamber and the dispersion liquid is frozen by a cooling unit located just below. It is equipped with a peltier cooling unit, and is possible to control accurate cooling temperature down to about -40 ° C. After freezing, vacuum drying treatment is performed with a dry pump. In this way, the dispersed state of nanoparticles is maintained. These procedures can be manipulated by GUI, and it is possible to prepare samples automatically and easily.

The dispersion state of particles by the freeze-drying method depends on the cooling temperature. If cooling is insufficient, "coffee ring" will be generated as shown in Fig. 2(a). Conversely, if excessive cooling is done, the particles will aggregate in the central area as shown in Fig. 2(c). When the nanoparticle dispersion is frozen at an appropriate temperature, the aggregated particles are extremely reduced and can be sufficiently dispersed as shown in Fig. 2(b). Accurate temperature control is important for appropriate specimen preparation.

This instrument can fix the particles only to a limited area on the substrate. If the particles are well dispersed, it is possible to count the number of all the particles contained in single droplet and measure each particle size by stitching observation within a realistic time frame (only several hours) as shown in Fig. 3. In this stitching observation, we conducted particle detection using image analysis.

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The detection ratio R is expressed by the formula R=D/N [%]. (D: number of particles counted by image analysis, N: total number of particles contained in a single droplet.). As a result, the detection rate R was 96%. This prototype is being improved for practical use [2].

References:
[1] K.Kumagai et al., 18th Int. Micro. Cong. Proceedings ID-5-P-1832 (2014), p. 3973.
[2] This research was supported by the "Bridging R&D and Business Promotion for Small and Medium-Sized Enterprises Project" in New Energy and Industrial Technology Development Organization (NEDO), Japan.

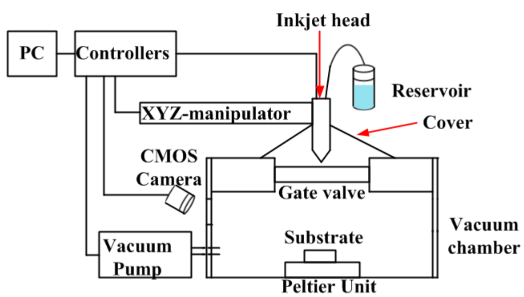
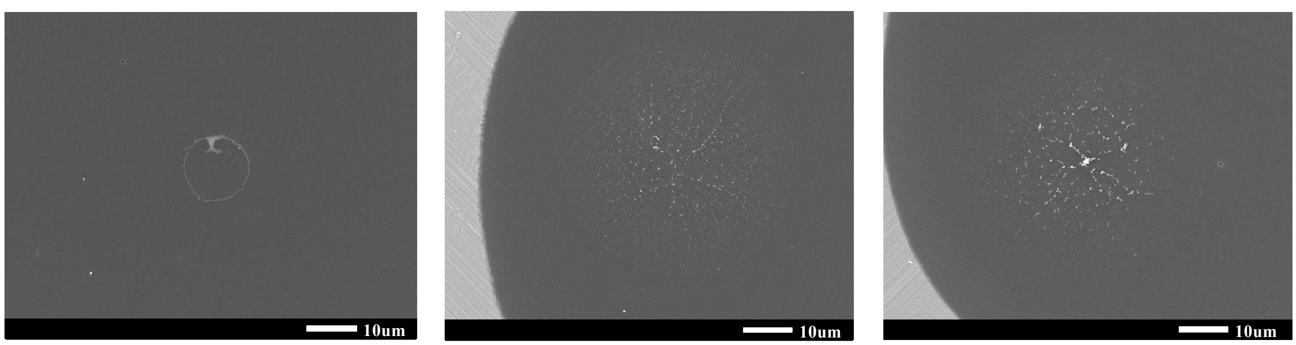


Figure 1. An overview of the automatic TEM/SEM specimen preparation instrument.



(a) High temperature (b) Appropriate temperature (c) Low temperature

Figure 2. The PSL spheres (100 nm dia.) spreading over a TEM support membrane. Temperature dependence can be confirmed for the state of dispersion.

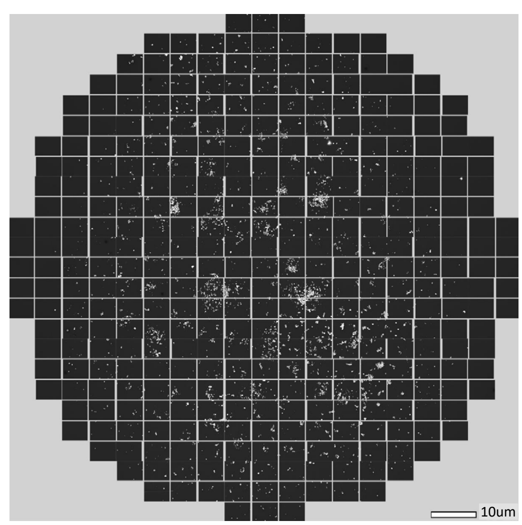


Figure 3. An example of stitching SEM observation over the whole area of a droplet.

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