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Facile *in situ* Lithiation and Sodiation Observation in TEM Employing MF (M=Li, Na)

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Various techniques were suggested for real time observation of alkali ion (Li and Na) storage into active materials in transmission electron microscope (TEM). Huang et al. employed a biasing holder for potentiostatic alkali ion storage [1]. Yuk et al. introduced graphene liquid cell TEM (GLC-TEM) [2-6]. However, the electrochemical holder requires a substantial expense for the holder, and sample preparation is relative inconvenient. Although GLC-TEM technique provides reducing environment for alkali ion storage in liquid electrolyte, alkali element amount in GLC is insufficient for complete reaction [2-6].

Hence, here, we report facile in-situ technique for alkali ion storage employing MF (M = Li, Na). To prepare TEM samples, active materials and MF are dispersed in ethanol. They are dropped on graphene-coated Au grid. MF particles easily decomposes by electron beam irradiation generating the alkali metals [7]. The alkali metals directly react with active materials [8]. The alkali ion storage process into an active material is well descripted in Figure 1. MF provides sufficient alkali metal for complete reaction. As example cases, we present lithiation and sodiation of CuS and perform comparison study between them (Figure 2). CuS experiences displacement reaction in lithiation forming copper dendrite outside crystalline Li_2S matrix. On the other hand, it forms Cu/Na_2S composite with uniformly distributed Cu in Na_2S matrix (Figure 2).

We believe this work provides invaluable insights for studying in-situ alkali ion storage mechanism into various active materials with the facile method [9].

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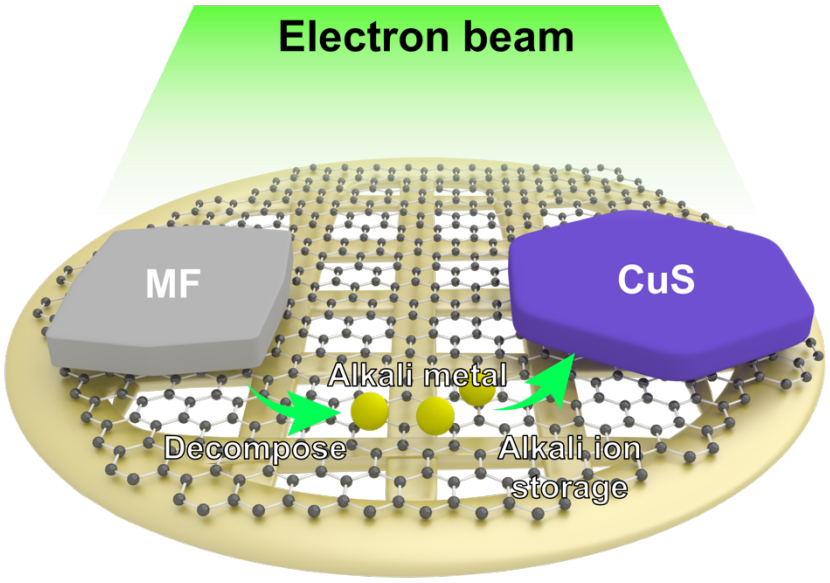

Figure 1. Schematic describing facile in-situ alkali ion storage method employing MF (M=Li, Na).

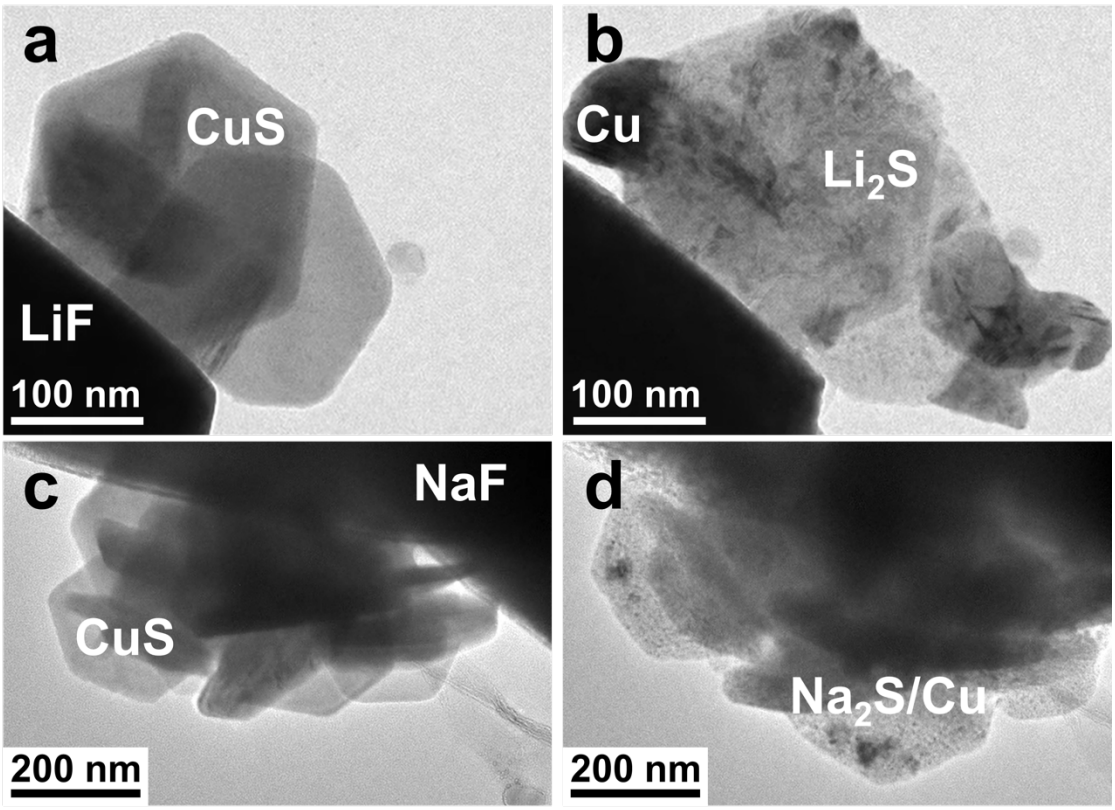

Figure 2. Example cases for lithium and sodium storages into CuS nanoplates. Low magnification TEM images of before (a) lithiation and (c) sodiation, and after (b) lithiation and (d) sodiation.

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