Article

Electrochemical solid-state amorphization in the immiscible Cu-Li system

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ABSTRACT

As a typical immiscible binary system, copper (Cu) and lithium (Li) show no alloying and chemical intermixing under normal circumstances. Here we show that, when decreasing Cu nanoparticle sizes into ultrasmall range, the nanoscale size effect can play a subtle yet critical role in mediating the chemical activity of Cu and therefore its miscibility with Li, such that the electrochemical alloying and solid-state amorphization will occur in such an immiscible system. This unusual observation was accomplished by performing in-situ studies of the electrochemical lithiation processes of individual CuO nanowires inside a transmission electron microscopy (TEM). Upon lithiation, CuO nanowires are first electrochemically reduced to form discrete ultrasmall Cu nanocrystals that, unexpectedly, can in turn undergo further electrochemical lithiation to form amorphous CuLi nanocrystals.

1. Introduction

Since the first discovery of amorphous metallic alloys in the early of 1960s, the past half a century has witnessed tremendous growth in both synthetic sophistication and depth characterization of this important class of materials [1–3]. Conventionally, amorphous metallic alloys can be synthesized by the rapid solidification of molten alloys, which is basically a physical method. As a fundamentally different scenario, the chemically driven solid-state amorphization that relies on the diffusive solid-state reactions at moderate temperature between pure metals has also been demonstrated early in 1983 as a viable approach for amorphous alloys formation [4]. More recently, it has been found that such a diffusive reaction based solid-state amorphization process can also be driven by electrochemical alloying at room temperature [5,6]. The most studied systems to date in the context of electrochemical solid-state amorphization are Li-Si and Li-Sn amorphous alloys [7,8]. Primary interest in these two systems stems from the potential utilizations of Si and Sn as high-capacity anode materials in Li-ion batteries. Both Li-Si and Li-Sn are typical miscible binary systems, with a negative heat of mixing ($\Delta H_{\text{mix}}$) as large as −30 and −18 kJ/mol [9], respectively. This large negative $\Delta H_{\text{mix}}$ provides the thermodynamic basis to enable chemical intermixing and amorphization through the diffusive solid-state reactions between Si (or Sn) and electrochemically generated Li [6]. In sharp contrast, however, Cu-Li is a well-known immiscible system with their $\Delta H_{\text{mix}}$ being near zero (0.56 kJ/mol by Calphad approach and −5 kJ/mol calculated by Miedema’s model) [9,10]. As such, Cu and Li have no or little tendency to spontaneously alloy on atomic scale in thermodynamic equilibrium [11,12].

A notable example that takes advantages of the immiscibility between Cu and Li is the widespread utilization of Cu foils as the anodic current collector in Li-ion batteries [13,14]. During operation of the battery, there exists a high concentration of electrochemically generated Li atoms at the Cu current collector surface, especially when the battery is fully charged. Owing to the equilibrium-immiscible nature of Cu with Li, the electrochemically generated Li atoms show no intermixing and alloying with the Cu current collectors (Fig. 1a), thereby assuring the long-term operation stability of Li-ion batteries [15]. Here we show for the first time that the nanoscale size effect can play a subtle yet critical role in mediating the chemical activity of Cu and therefore its miscibility with Li. When decreasing nanoparticle sizes into ultrasmall range, Cu as a parent phase can react readily with the electrochemically generated Li, thereby leading to the occurrence of diffusion alloying and solid-state amorphization in such an equilibrium-
immiscible binary metallic system (Fig. 1b). It is the dramatically enhanced surface free energy of the ultrasmall Cu nanoparticles that constitutes the thermodynamic basis for driving electrochemical lithiation and amorphization.

2. Experimental

Our work was accomplished by taking advantage of an in-situ transmission electron microscopy (TEM) platform that allows for the real-time studies of nanoscale electrochemical cells under dynamic operating conditions. Experimentally, a solid-state open cell configuration (Fig. S1 online) was adopted for in-situ TEM studies, with the selected individual CuO nanowire (fixed on a gold tip) as the working electrode, bulk Li metal (fixed on the tungsten tip) as the counter electrode, and the naturally grown thin Li2O layer on Li metal as the solid-state electrolyte [16]. All the components were loaded onto a TEM specimen holder (Picofemto) integrated with mobile electrical biasing probe. A piezo-driven nanomanipulator was used to manipulate the tungsten tip to contact a selected CuO nanowire and then a bias of 3–5 V was applied on Li electrode with respect to CuO electrode to drive Li+ transport through the solid-state Li2O layer. In this way, the electrochemical lithiation of CuO nanowire was initiated (Fig. S2 online, see Supporting Information for more experimental details).

3. Results and discussions

Upon lithiation, CuO nanowires were electrochemically reduced to discrete ultrasmall Cu nanoparticles that are embedded in Li2O matrix (Fig. S3 online), as consistent with that reported in our previous work [17] and the related work by Golberg’s group [18]. Here we denote this lithiation process (CuO + Li+ + e− → Cu + Li2O) as the “1st stage” lithiation. An intriguing new finding in our present study is the observation that, following the “1st stage” lithiation process, there will occur a previously unknown “2nd stage” electrochemical lithiation process, i.e., the as-formed ultrasmall Cu nanoparticles embedded in Li2O matrix can be further electrochemically lithiated to form amorphous CuLi_x nanoalloys (Cu + Li+ + e− → CuLi_x).

In Fig. 2, we show how the two stages of lithiation processes occur in sequence. As the “1st stage” lithiation reaction proceeds, a clear reaction front that separates the lithiated phases from the pristine CuO phase can be clearly seen in the nanowire (Fig. 2a). With the occurrence of “2nd stage” lithiation, a new reaction front will emerge, along with the first one; as a result, the nanowire is separated into three distinct segments (Fig. 2b). A continuous movie that recording the entire lithiation processes is shown in Movie S1 (online). Interestingly, it can be noticed that the newly formed “2nd stage” lithiated region exhibits a much lighter image contrast, which is reminiscent of the possible formation of some amorphous phases. To confirm this, we comparatively probed these two lithiated regions with selected area electron diffraction (SAED). Whereas SAED pattern of the “1st stage” lithiated region shows the clear presence of diffraction rings both from Li2O and Cu, SAED pattern of the “2nd stage” lithiated region displays only the diffraction rings of Li2O, as expected. Taken together, the observed distinct contrast fading of TEM image and the disappearance of Li diffraction rings thereby confirm that the nanocrystalline Cu particles were amorphized during the “2nd stage” lithiation process.

A key question then arises: is this amorphization event caused by simple crystalline-to-amorphous transformation of Cu nanoparticles themselves, or otherwise, by lithiation-induced solid-state amorphization of nanocrystalline Cu particles, just like what happens in Li-Si and Li-Sn systems. In our present work, we have got multiple complementary experimental evidences to validate that this amorphization event is due to the latter case. The first evidence comes from scanning transmission electron microscopy high-angle annular dark field (STEM-HAADF) imaging results where the formation of Cu-Li alloying phase can be identified. Electron energy loss spectroscopy (EELS) characterization further reveals that there is a net charge transfer from Li to Cu, a straightforward evidence of the alloying between Cu and Li. Moreover, real-time kinetic measurements of the “2nd stage” lithiation process also unveil a very slow reaction rate that is typical of the solid-state diffusive alloying reactions. The last evidence comes from a remarkable finding that the deliberate irradiation of the as-formed amorphous CuLi_x nanoalloys by electron beam can induce the delithiation of them, i.e., the amorphous alloying phenomena is reversible in nature.

Fig. 3a displays a representative STEM-HAADF image that depicts a comparison between the “1st stage” and “2nd stage” lithiated regions in a same nanowire, where a distinct difference of image intensity can be seen. This is due to the atomic-number (Z) sensitive nature of HAADF contrast (Z-contrast) and the fact that the atomic
number of Cu is far larger than that of Li. Therefore, the substantially
reduced HAADF image intensity in the “2nd stage” lithiated region
indicates the formation of a “diluted Cu” phase, a key evidence that
the ultrasmall Cu nanoparticles have been lithiated to form the CuLi
alloying phases during the “2nd stage” lithiation process. An
enlarged higher-resolution STEM-HAADF image is shown in
Fig. 3b, where the individual Cu and lithiated CuLi nanoparticles
can be discerned. From the corresponding STEM-bright field (BF)
image (Fig. 3c), the lattice fringes of Cu nanoparticles in the “1st
stage” lithiated area are clearly resolved whereas the nanoparticles
in the “2nd stage” lithiated area show no lattice fringes under
the same focus condition, indicating that CuLi alloying phase is of
an amorphous nature. This result is in well consistence with the
SAED results as discussed above and the high-resolution TEM
(HRTEM) imaging results of the “2nd stage” lithiated region (Fig. S4
online).

Fig. 3d shows a comparison of Cu-L2,3 EELS spectra of the CuO
nanowire, the ultramall Cu nanoparticles dispersed in Li2O matrix
and the amorphous CuLi nanoalloys, respectively. For transition
metals, L2,3 edge arises through dipole transition from core-level
2p electrons to the narrow unoccupied d states. Normally, two
sharp peaks known as “white lines” can be observed at the onsets
of the L2 and L3 absorption edges, the intensities of which show
strong correlations with d-state occupancy [19]. In the case of ele-
mental Cu, where there are no unfilled d states (Fig. 3e), no sharp
white lines can be observed, leaving only steps in L2,3 absorption
edge [20]. In the case of CuO, the electron transfer from the Cu
3d orbital to oxygen will lead to the appearance of white lines
together with a slightly lowered EELS threshold energy of the L3
edge, as seen in Fig. 3d. As far as Cu-based alloys are concerned,
the situation is much more complicated. When alloying with early
transition metals with low d-state occupancy, such as in Cu-Zr and
Cu-Ti, EELS spectra are featured by a large enhancement of the
white line intensities, indicating that there is a net d-electron
transfer from Cu to Zr or Ti [21]. On the contrary, when alloying
of Cu with typical electron-donating metals, such as Cu-Li system
in our case, one can expect that charge transfer will occur from
Li to Cu, which will increase the overall occupancy of Cu 3d–4s
band (Fig. 3e) in the as-formed CuLi alloys and thereby diminish the intensity of L_{2,3} absorption edge. As shown in Fig. 3d, the spectrum feature of amorphous Cu-Li nanoalloys is well consistent with these expectations, although it is difficult to quantify the amount of transferred charge.

As compared to the “1st stage” lithiation, the “2nd stage” lithiation proceeds much slower. This is reasonable, considering the fact that the solid-state Cu-Li alloying reaction involves a sluggish diffusion process, that is, the slow diffusion of the freshly formed Li atoms into the lattice of pre-formed Cu nanoparticles[22,23]. From the time-lapse TEM images in Fig. 4a, we can clearly see the slow propagation of the reaction front during the “2nd stage” lithiation process. In-situ kinetic measurement was made, and a plot of L (the moving distance of the “2nd front” compared to the position of it at the selected time of 0 s) versus time t, is shown in Fig. 4b where the deduced reaction rate \( \approx 0.3 \) nm/s. It is important to note that such a slow reaction rate is common for all of the tested nanowire samples (Fig. S6 online), although the deduced values vary a little from sample to sample. As a whole, the reaction rate of “2nd stage” lithiation is around one order of magnitude slower than that of the “1st stage” lithiation [17]. Moreover, the nearly perfect linear relationship between L and t (Figs. 4b and S6 (online)) implies that the electrochemical alloying of Cu-Li is a reaction limited process. In other words, the slow diffusive Cu-Li alloying reaction that occurs at a rate limited by Li diffusion in lattice of Cu nanoparticles, is a decisive step over the “2nd stage” lithiation process.

A key determinant of driving a solid-state amorphization process, either chemically or electrochemically, is to elevate the free energy of the pure reactants to an energy level being higher than that of the metastable amorphous alloying phases. For systems with a large negative heat of mixing, such as Li-Si and Li-Sn, this thermodynamic requirement is relatively easier to fulfil, and therefore the solid-state amorphization reaction can more readily occur. By contrast, for an immiscible system with less negative or even positive heat of mixing, such as Cu-Li, the occurrence of solid-state alloying and amorphization is less likely under normal circumstances. Nevertheless, by purposely utilizing the nanoscale size effects to impart excess surface/interface free energy, the free energy of reactants can be elevated to high “ladder” such that the amorphous alloying becomes possible [24,25]. In our present work, the Cu nanoparticles resulting from the in-situ reduction of CuO nanowires during “1st stage” lithiation, have got an ultrasmall size region focusing around 2–5 nm (Fig. 4c). This extremely ultrasmall size marks the point at which the majority of atoms in a specific Cu nanoparticle are at its surface [26], which can dramatically increase the surface free energy of the system compared to bulk Cu or larger crystal size Cu [11,27,28]. Therefore, it is the nanoscale size effect that plays a subtle yet critical role in mediating the chemical activity of Cu and therefore its miscibility with Li, which allows for the occurrence of electrochemical alloying and amorphization in such an immiscible system. Interestingly, with a close examination of the time-lapse images in Fig. 4a, a fact that should not be ignored is that, although the large majority of Cu nanopar-
Particles were transformed to amorphous CuLi\textsubscript{x} nanoalloys, there still are some larger-sized Cu nanoparticles being left behind the reaction front. This implies that there exists a critical size above which the nanocrystalline Cu particles cannot be lithiated to form amorphous phases, a key manifestation of the nanoscale size effect. From the comparative statistic results shown in Fig. 4c, the critical grain size can be experimentally identified to be ca. 6 nm. Moreover, we also carried out control experiment with the generated larger Cu nanoparticles (Fig. S7 online) to verify the nanoscale size effect and existence of critical size.

For in-situ TEM studies, the effect of the imaging electron beam irradiation requires careful evaluation. In our present work, we have carefully examined whether or not there is possibility that the occurrence of Cu-Li amorphization was induced by the electron beam irradiation. When the electron beam was deliberately blanked or spread to be extremely weak (Fig. S8 online), it was found that the solid-state Cu-Li alloying and amorphization remained to occur. Therefore, it is safe to say that this process is exactly electrochemically driven, rather than due to electron beam irradiation effect. On the contrary, it was surprisingly found that the electron beam irradiation could actually induce an opposite result. That is, under intense electron beam irradiation, the as-formed amorphous CuLi\textsubscript{x} alloys would be delithiated, accompanied with the transformation of amorphous CuLi\textsubscript{x} alloys to metallic Cu nanoparticles (Fig. 5). Here it is worth noting that the similar electron-beam-induced-delithiation phenomenon had also been previously observed by Wang [29] when studying the lithiation of Si nanowire. Although the underlying mechanism of the electron beam induced dealloying phenomenon remains elusive, this remarkable observation anyhow demonstrates that the possibility that the observed solid-state amorphization of Cu-Li might be induced by electron beam irradiation effect can be explicitly ruled out.

Fig. 4. Lithiation kinetics and nanoscale size effect in Cu-Li electrochemical alloying and amorphization. (a) Time-lapse TEM images showing the propagation of the reaction front during “2nd stage” lithiation (see also Movie S2 (online)). The yellow arrow in each frame indicates the “2nd front”. Scale bar, 50 nm. (b) A plot of distance \( L \) (the “2nd stage” reaction front propagation length) versus the corresponding time \( t \). (c) The statistical particle size distribution of the unalloyed Cu nanoparticles (top) and the pristine as-formed Cu nanoparticles (bottom), correspondingly measured from the area framed in (a) by the red rectangle and the HRTEM image in Fig. S3 (online) of the blue rectangle framed area.
Chinese Academy of Sciences for fruitful discussions about solid-state amorphization.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.scib.2018.06.021.

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