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# Three-dimensionally interconnected nickel-antimony intermetallic hollow nanospheres as anode material for high-rate sodium-ion batteries

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**Abstract**
Three-dimensionally interconnected NiSb intermetallic hollow nanospheres for Na-ion batteries are prepared by a facile and low-temperature approach involving crystallized 3D interconnected Ni nanospheres precursor and subsequent galvanic replacement reaction involving Sb ions. The 3D interconnected hollow structure and Ni matrix encapsulation contribute significantly to the excellent structure stability and high electrochemical performance of the Sb-based anode. They exhibit highly stable and substantial discharge capacities of 400, 372, 230 mA h g$^{-1}$ after 150 cycles at 1C, 5C, 10C, respectively. Moreover, a full $Na_{0.4}Mn_{0.54}Co_{0.46}O_2$//NiSb battery shows at a current density of 300 mA/$g_{Sb}$ a charge and discharge capacity of 451 and 301 mA h g$^{-1}$, respectively, and also displayed relatively good stability, retaining 75% of the initial discharge capacity after 20 cycles.
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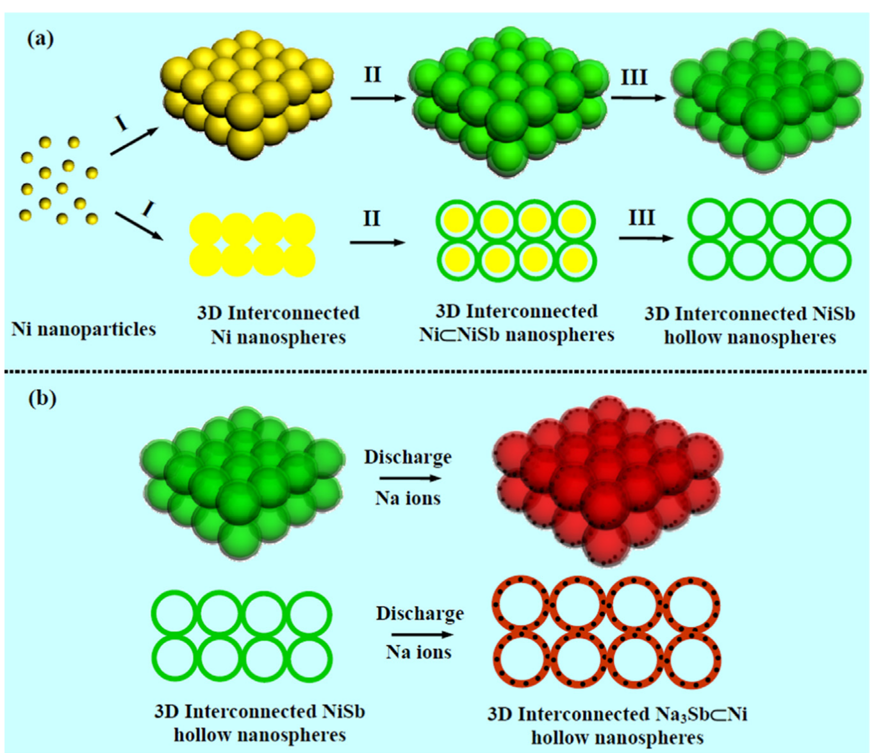
Lithium-ion batteries have become one of the most important devices for energy storage and are now industry standards for powering electric/hybrid electric vehicles and for renewable energy storage [1-6]. However, considering the limited and unevenly distributed availability of Li deposits, cost will become an increasing issue for Li-ion batteries; while sodium-ion batteries have the advantage of a much greater Na-abundance [7,8]. The larger size by about 55%, the larger weight and the less favorable redox potential, though, are normally disadvantages of a high performance Na storage [9]. Nevertheless substantial attention has been paid to Na-ion battery cathode materials in recent years, and progress has been leading to the development of $Na_{0.85}Li_{0.17}Ni_{0.21}Mn_{0.64}O_2$, $Na_4Mn_9O_{18}$, $Na_{0.6}Fe_{0.5}Mn_{0.5}O_2$, $Na_4Fe(CN)_6$, or $Na_3V_2(PO_4)_3$ [10-16]. In particular $Na_3V_2(PO_4)_3$ has been shown to even outperform Li-ion battery cathodes in terms of capacity per weight owing to excellent kinetic properties [16]. As far as anodes are concerned, earlier works devoted to Ti-based materials and carbonaceous materials such as carbon hollow nanospheres, showed that they deliver a reversible capacity of approximately 250–300 mA h $g^{-1}$ with reasonable cycling stability [17-19]. Concerning carbon structures, further capacity increase can not be expected due to limitation of sodium host sites. Besides, the sodium insertion potential in carbonaceous anode materials is very close to the deposition potential sodium, which can cause a severe safety issue when a slight polarization occurs. Na alloy-based anodes can provide appropriate sodium insertion potentials and much higher gravimetric and volumetric specific capacities compared to carbonaceous materials, such as Sn ($Na_{15}Sn_4$, 847 mA h $g^{-1}$) and Sb ($Na_3Sb$, 660 mA h $g^{-1}$) [20-25,26-28]. However, the rapid capacity decay of such anodes originated from the large volume changes severely hinder their applications for Na-ion batteries.

Much research effort has been devoted to overcome this problem in previous research work [20-34]. A promising approach is to move to M-Sn/Sb intermetallics [25,31,34], that convert in the first cycle to M:Sn/Sb composites. The primary beneficial role of the metal M is to provide a mechanical buffer for accommodating volume changes that otherwise would lead to disintegration [25]. Three dimensional (3D) uniform porous or hollow nanoarchitectures such as 3D porous foam and 3D interconnected hollow nanospheres are generally considered as high-capacity and stable electrode materials, due to their uniform microstructure, 3D electronic conduction and especially pronounced tolerance to stress change [35-37]. Compared with the Sn-based alloy anodes, the Sb-based anode have much higher microstructural flexibility as the melting point of Sb (631 °C) is much higher than that of Sn (232 °C). Herein, we report a low-temperature and facile synthesis of a new type of nanoarchitectured Ni-Sb intermetallic anodes, viz., 3D hierarchical interconnected microstructures composed of monodisperse zero dimensional (0D) hollow nanospheres units. The well-defined hollow cores of nanospheres and interconnected pores can also provide additional elastic




**Scheme 1** (a) Schematic illustration of the fabrication of 3D interconnected NiSb hollow nanospheres anode for Na-ion batteries: (I) uniform magnetic Ni nanoparticles behave as single magnetic dipole, self-assembling Ni nanospheres for 3D interconnected structures in solution because of strong magnetic dipolar interactions; (II, III) 3D interconnected NiSb intermetallic hollow nanospheres formed by a subsequent galvanic replacement process with such 3D interconnected Ni nanospheres as the template and precursor; (b) schematic representation of the first sodiation of 3D interconnected NiSb intermetallic hollow nanospheres, forming 0D electroactive $Na_3Sb$ nanoparticles embedded in 3D conducting Ni hollow matrix.

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