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Structural and electrochemical performance of Al-Mo Thin film alloy anodes for Lithium ion Micro Batteries

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Abstract: The Al_5Mo thin film anodes for Li ion battery were prepared using DC sputtering method in different conditions like deposition at room temperature (S0) and deposition at 300ºC (S1). The thin films were deposited from Aluminum target tiled with Molybdenum discs in the ratio calculated on the basis of theoretical sputtering yield. The structural analysis performed using XRD had confirmed the Al_5MO compound formation in rhombohedral geometry. The compound formation was observed to be evident only for the thin films when subjected to heat treatment while deposition. The scanning electron micrographs of the samples had revealed higher porosity around 23% for S0 sample and lower porosity around 18% for S1 sample. The chrono-potentiometry results of the samples in non aqueous electrolyte versus Lithium counter electrode had shown higher volumetric specific capacity around 197 mAh/cm^3 for S1 sample. The capacity increment had been observed for all the samples upon charge-discharge cycling and the extent of increment after 25 cycles for S0 and S1 samples were observed to be 41.2% and 20.4% respectively.
Keywords: Thinfilm anode; X-ray Diffraction; Scanning Electron Microscopy; Cycle life study.

Introduction and Experimental

In most of the portable electronic equipment, Li ion secondary batteries are widely been deployed as power sources. Despite the implication of nano material electrodes, the extent of miniaturization of conventional Li ion batteries remains limited. At this juncture a research group led by J.B. bates and Nancy dudney et al from ORNL demonstrated solid state thin film Li ion batteries which attracted remarkable attention in late 90's [1,2]. The thin film batteries are viewed as futuristic power source by several researchers due to its efficacious performance and compatibility with IC components. In general, the theoretical specific capacities of the Li alloys are higher than the widely used lithiated graphite anode and slightly lesser than the metallic Lithium anode [3]. Widely investigated metal anodes include Al [4], Sn [5], Al-Sn alloy [6] and so on. Despite many advantages, the abnormal volume expansion while charging is observed to be common for all the metal anodes [7-9].Hence in this paper the inclusion of electrochemically inactive Molybdenum (Mo) in to the Aluminum (Al) matrix has been investigated with the idea of alleviating the irreversible expansion occurring in the aluminum while charging.

The Al-Mo intermetallic alloy anode thin films were prepared on copper substrate by DC magnetron sputtering process. The sputtering target was developed by new tiling method. The stoichiometric alloy was

prepared by sputtering yield ratio calculation. Al_5Mo thin film was deposited in Cu substrate. The sputtering deposition from the target was achieved by applying the power of 80 W and the films were coated to the approximate thickness of 2 µm. The thin film depositions were carried out with and without substrate heating upto 300ºC. The structural analysis of the processed film was carried out by Philips PW 1710 diffractometer and the parameters were compared with standard JCPDS data. The morphological characterization of the film was analyzed by using JEOL, JSM – 6460 scanning electron microscopy. The electrochemical studies of the thin films of area $1cm^2$ were studied by micro autolab FRA III in non aqueous electrolyte (1M $LiPF_6$ dissolved in 1:1 ratio of EC and DMC respectively) Vs Li foil. The charge discharge analysis was carried out with the potential window of 2.1V to 3V at the current density of $\pm 100 \mu A/cm^2$.

Results and Discussion

The X ray diffractogram of thin film samples (i) deposited at room temperature without substrate heating (S0), (ii) deposited at 300ºC with substrate heating (S1) are represented in Figure 1 and it is evident that the deposition in room temperature has not yielded crystalline thin films of either Al, Mo or their intermetallics. This may be attributed to the lack of nucleation in the cold substrate. Whereas, the samples S1 have shown characteristic peak of Al_5Mo around 43.1 degrees (ICDD file No.65-1144) corresponding to (024) plane. The Cu substrate is observed around 50.18 degrees (ICDD file no. 89-2838) corresponding to (200) plane for both samples. Apart from the Al_5Mo and Cu substrate peak, another peak corresponding to Mo has occurred in sample S1.This may be attributed to the precipitation of smaller volume of Mo in Al_5Mo matrix.

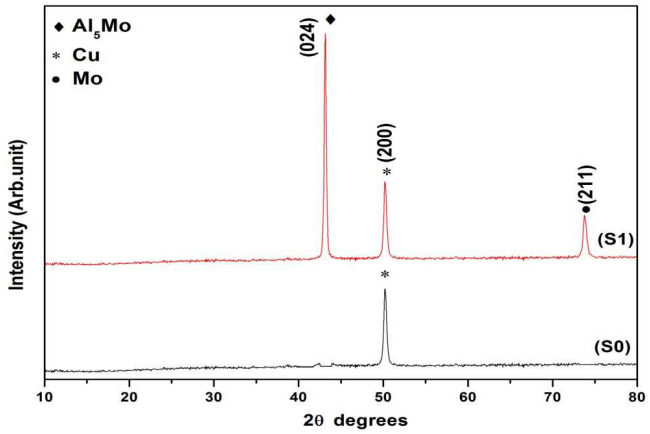

Figure 1: Structural analysis of deposited at room temperature (S0) and deposited at substrate heating (S1)

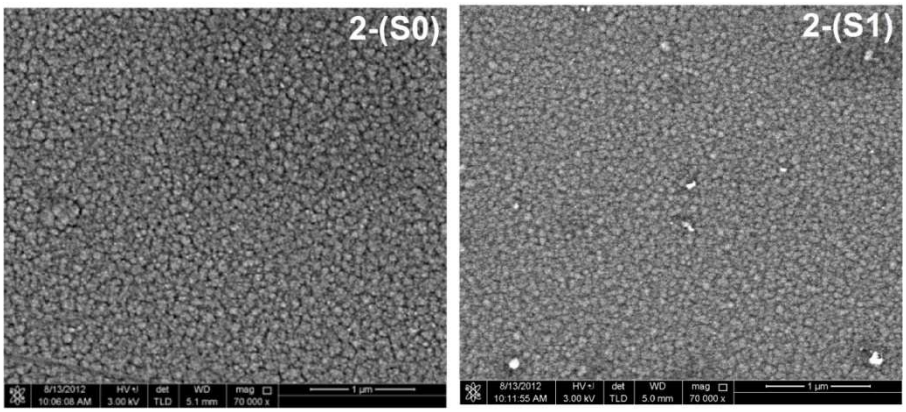

Figure 2: Morphological analysis of deposited at room temperature (S0) and deposited at substrate heating (S1)

The scanning electron micrograph of the samples S0 and S1 are presented in Figure 2. From the figure, it could be observed that all the films possess homogeneously distributed smaller grains without significant grain agglomeration. The Figure 3 illustrates the charge discharge cycle study of samples S0 and S1 respectively. From the graph, the volumetric charging capacity of the samples could be calculated to be around 109.0 mAh/cm^3 and 196.6 mAh/cm^3 for the samples S0 and S1 respectively. The charge and discharge capacity are observed to be higher for S1 sample which has been deposited in the temperature of 300ºC using substrate heater. It may be due to the homogeneous distribution of Mo in Al_5Mo matrix.

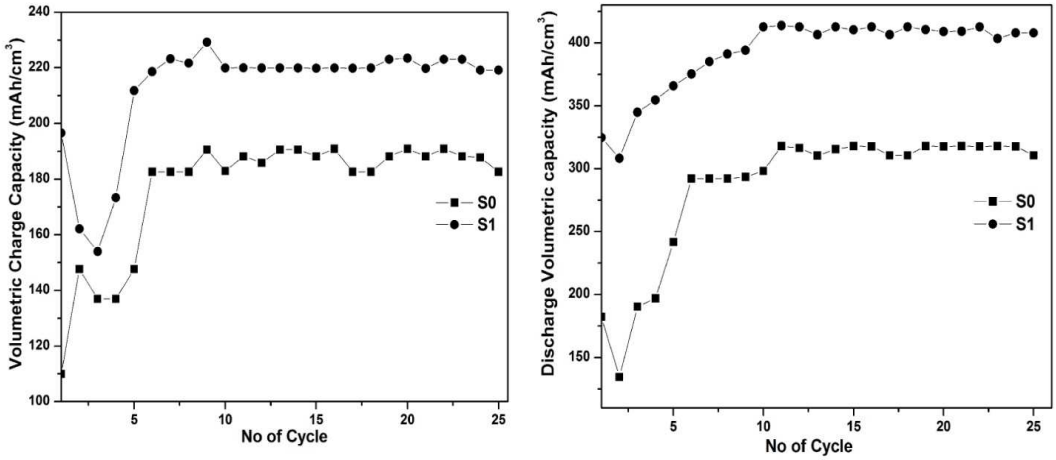

Figure 3: charge discharge Cycle life study of Al_5Mo alloy thin films

From the figure 3, it could be observed that specific charge/discharge capacity increases for all the samples after 25 cycles. This may be attributed to the gradual increase in diffusion of Li ions into Al_5Mo matrix. The electrochemically inert Mo decreases the characteristic anomalous volume expansion of Al.The percentage discharge capacity increment after 25 cycles for the electrodes are observed to be around 41.2%, 20.4% and 21.1% for samples S0 and S1 respectively.

The preparation of Al-Mo alloy thin film from through DC sputtering from indigenously tiled target has been exemplified in this manuscript. The X-ray diffraction results have further revealed the crystalline compound formation only due to the heat treatment during the deposition. From the Electrochemical Studies it could be concluded that the S1 sample possess higher volumetric discharge capacity and which may corroborated to the homogeneous film formation due to heating while deposition. This work is actually intended to alleviate the anomalous irreversible volume expansion of Al anode occurring while charge- discharge cycles by the inclusion of electrochemically inactive Molybdenum (Mo) in to the Aluminum (Al) matrix.

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