Journal of Energy Storage 8 (2016) 91–98

Contents lists available at ScienceDirect

Journal of Energy Storage

journal homepage: www.elsevier.com/locate/est

The effect of felt compression on the performance and pressure drop of all-vanadium redox flow batteries

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ARTICLE INFO

Article history:
Received 9 August 2016
Received in revised form 2 October 2016
Accepted 5 October 2016
Available online 17 October 2016

Keywords:
Energy storage
X-ray computed tomography
Flow
Contact resistance
Compression

ABSTRACT

The compression of carbon felt electrodes for redox flow batteries leads to changes in the electrochemical performance and has a large effect on the pressure drop of electrolyte flow through the system. In this investigation, the authors have characterised the electrochemical performance of all-vanadium redox flow batteries by studying the effect of compression on the contact resistance, polarisation behaviour and efficiency. Contact resistance was seen to reduce from *ca.* 2.0 Ω cm^2 to 1.2 Ω cm^2 and an energy efficiency of 85% was obtained from a felt compressed to 75%. Moreover, X-ray computed tomography (CT) has been employed to study the microstructure of felt electrodes at compressions up to 70%, showing a linear decrease in porosity and a constant fibre surface area-to-volume ratio. The pressure drop was modelled using computational fluid dynamics and employing the 3D structure of the felts obtained from CT, revealing that a 60% increase in compression related to a 44.5% increase in pressure drop.

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1. Introduction

Redox flow batteries (RFBs) are promising candidates for a variety of large scale energy storage solutions, such as optimising renewable energy sources with intermittent supply, standalone power systems, frequency regulation and peak shaving applications. RFBs have a particular advantage of being able to de-couple power from energy: the former is a characteristic of the cell(s) architecture (electrode area, operating voltage, etc.), whereas the latter is dependent on the volume (and concentration) of electrolyte contained in external storage tanks. This gives RFBs great design flexibility to match the requirements of a range of applications. Although many RFB chemistries have been reported, including bromine-based [1–3], iron-based [4,5] and organic-based systems [6–8], the most developed is the all-vanadium redox flow battery (VRFB) [9]. The VRFB utilizes the four stable oxidation states of vanadium: on the negative electrode vanadium exists in the V^{2+} (charged) and V^{3+} (discharged) states, whilst on the positive electrode vanadium exists in the V^{5+} (charged) and V^{4+} (discharged) states. An advantage of RFB systems over traditional battery systems is the rate of self-discharge may be more easily controlled by removal of reactants from the reaction area of the cell when the battery is not in use. Flow batteries can suffer from reactant cross-over, the rate and selectivity of which is determined by the nature of the separator employed, which reduces the capacity of the battery. However, an advantage of the all-vanadium system, over other mixed flow battery chemistries, is that any imbalance from cross-over is able to be addressed electrochemically: *i.e.*, the electrolyte can be electrochemically regenerated without any chemical separation of reactant required, by virtue of the reactants all being the same chemical species. However, RFBs, in general, tend to suffer from low energy and power densities compared to hydrogen fuel cells and Li-ion batteries.

Many studies have attempted to improve the performance of RFBs by altering numerous design aspects of cells and stacks. Examples include thermal and chemical treatments of electrodes and separators [10,11], electrode modifications [12,13], perforating electrodes to decrease pressure drops [14] and addition of chemicals to the electrolyte [15,16]. One particular study investigated the effect of mechanical, electrical and morphological properties on compressed electrodes in VRFBs [17]. The study showed that an increase of cell clamping pressure increases the peak power outputs of RFB systems. The study only considers felts compressed to a maximum of 40% and the method of extracting the

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http://dx.doi.org/10.1016/j.est.2016.10.003
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porosity of these electrodes is based on a 2D optical technique containing many assumptions, which lead to an estimated porosity.

Microstructural properties such as porosity and tortuosity of electrodes are inherently 3D properties and should therefore be studied in three dimensions. Microstructural characteristics of RFB electrodes are significant as they may have a large effect on the performance of said electrodes; for example, electrolyte intrusion, pressure drop and porosity. Jervis *et al.* have designed a flow cell which permits *in situ* microstructural characterisation of flow battery innards in three dimensions [18]. In this study, the authors expand on the work of Chang *et al.* by considering felts over a broader compression range and using X-ray computed tomography (CT) to extract true values of the microstructure of felt electrodes (porosity, surface area, *etc.*) in three dimensions. Moreover, by reconstructing the actual felt structures in three dimensions, computational fluid dynamics (CFD) may be used to model the effect of felt compression on the liquid pressure drop through the actual structure of the felts to deduce how the orientation of the felt fibres under compression affects the electrolyte flow.

2. Experimental

2.1. Electrochemical characterisation

Carbon felts with an uncompressed thickness of 3.18 mm (99% carbon, Alfa Aesar (43199.RR)) were heat treated in air at 400 °C for 30 h to thermally activate the fibre surfaces, as described by Sun *et al.* [10]. The felts were then cut into $5 \times 5\ cm^2$ for electrochemical testing and into 1/8″ discs for the X-ray tomography studies using a hollow punch, which is described below. Electrolyte membrane (Nafion 117) was cut using a CO_2 laser and then treated in 3% H_2O_2 for one hour, followed by thermal treatment in 1 M H_2SO_4 at 80 °C before being rinsed in de-ionised water and oven dried at 60 °C for 30 min. Graphite composite blocks with a triple serpentine flow field (1 mm channel width, 1 mm depth) were fabricated in-house using a CNC routing machine; these components acted to help distribute the anolyte and catholyte to the felt electrodes and conduct charge to the external circuit via gold coated copper plates. PTFE spacers of various thicknesses were used to control the compression of the felt electrodes. The whole assembly was held together using bolted end-plates by tightening the eight bolts to a torque of 12 Nm and is shown in Fig. 1(a). The electrolyte used was a 1.6 M vanadium species (50:50 ratio V^{3+}:V^{4+}) in 3 M H_2SO_4 (PV3 Technologies, UK). A three-arm peristaltic pump (Watson-Marlow Ltd., UK) was used to pump the electrolytes at a normalised flowrate of 2 mL min^{-1} per cm^2 of electrode area. The catholyte and anolyte were kept under a flow of nitrogen and the battery was charged at 50 mA cm^{-2} until a potential of 1.6 V was reached before holding potentiostatically at 1.6 V until the current fell below 2 mA cm^{-2}. Once charged, electrochemical impedance spectroscopy was performed to deduce the high frequency intercept at open circuit (representing the Ohmic resistance of the cell) before a galvanostatic polarisation curve was performed. Charge-discharge cycling was performed at a current density of 50 mA cm^{-2} until cell voltage limits of 1.6 V and 1.0 V were reached, respectively. Electrical resistivity measurements were performed on a single, heat treated, SGL GFA6 felt and used the same setup but without electrolyte flow and a membrane. All electrochemical measurements were made using an Ivium NStat potentiostat (±5 A, ±12 V (Ivium Technologies B.V., NL)) apart from the electrical resistivity measurements which were obtained from a Gamry Reference 3000 equipped with a Gamry 30k Booster (Gamry Instruments, USA).

2.2. X-ray computed tomography and pressure drop modelling

Three 1/8″ diameter tokens were cut from the carbon felts and then assembled into a fibreglass housing with various thickness spacers, used to control the compression. The housing was laminated with a fibreglass cap with the use of epoxy and held under pressure for 24 h to allow the epoxy to cure. The resulting tokens containing the compressed felts (assembly shown in Fig. 1(b)) were then placed inside a ZEISS Xradia Versa 520 3D X-ray microscope (Zeiss, Pleasanton, CA). The tokens were scanned using the laboratory X-ray source utilising a tungsten target and acceleration voltage of 40 kV_p. A total of 1601 projections were taken with 12 s exposure over 360° using an objective lens with 4× magnification, resulting in a pixel size of 3.48 μm.

The tomograms were reconstructed using a filtered back-projection algorithm (Zeiss XM Reconstructor) and imported into the Avizo software suite (FEI Co, USA) in order to calculate the porosity of the samples and to measure the exact compression of each of the tokens. Smaller, representative volumes of 1 mm^3 of each of these scans were then cropped from the original data.

A surface mesh was generated for the three felts and contained 2.4×10^6, 2.9×10^6 and 3.8×10^6 mesh elements respectively (the increase in the number of mesh elements is due to more fibres being present in the sample volume with compression). The resulting meshes were imported into the StarCCM+ computational fluid dynamics software suite (CD Adapco, UK) [19–21]. This enabled modelling of the pressure drop through the samples to be achieved. A Boolean subtraction of the fibre volume was used in

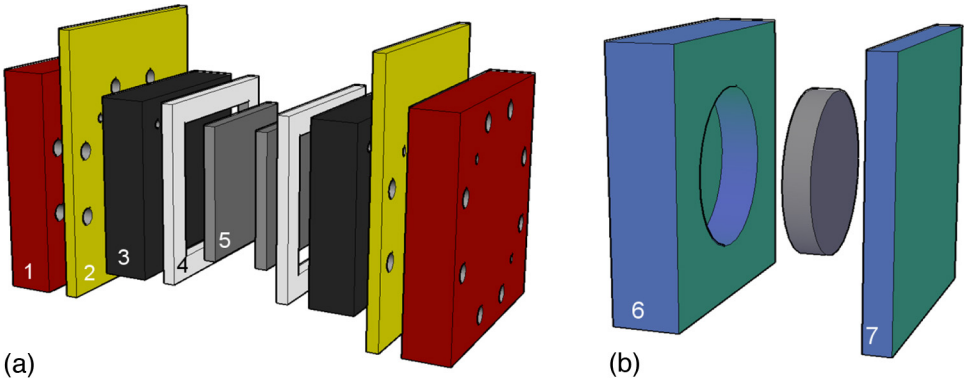

Fig. 1. Illustration showing: (a) the cell assembly for experimentation containing: 1 the bolted end plates; 2 the gold plated copper current collectors; 3 the POCO graphite composite flow field plate (flow fields not shown); 4 the PTFE spacers used to control compression and 5 the felt electrode. The membrane is not shown. (b) Shows the fiberglass housing (6). The thickness of the housing was changed in order to control the compression on various felts. The fibreglass lid is also shown (7).

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