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### Title

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### **Development of a Li<sup>+</sup> alumino-silicate ion source<sup>\*</sup>**

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#### Abstract

To uniformly heat targets to electron-volt temperatures for the study of warm dense matter, one strategy is to deposit most of the ion energy at the peak of energy loss (dE/dx) with a low (E <5 MeV) kinetic energy beam and a thin target. Lower mass ions, such as lithium, have a peak dE/dx at a lower kinetic energy. Alumino-silicates sources of K<sup>+</sup> and Cs<sup>+</sup> have been used extensively in beam experiments, but there are additional challenges for the production of a high current and high-quality Li<sup>+</sup> beam. There are tighter tolerances in preparing and sintering the Li aluminosilicate to the substrate to produce an emitter that gives uniform ion emission, sufficient current density and low beam emittance. Here we report recent progress on Li<sup>+</sup> source studies.

#### **INTRODUCTION**

To uniformly heat targets to electron-volt temperatures for the study of warm dense matter with intense ion beams [1], low mass ions, such as lithium, have a peak dE/dx at a lower kinetic energy. Traditionally, Li<sup>+</sup> beams have been produced by thermionic emission from aluminosilicates compounds  $\beta$ -spodumene and  $\beta$ -eucryptite [2]-[11]. Though  $\beta$ -spodumene is much more stable, its ion emission is almost half of the  $\beta$ -eucryptite. The CW current extraction from  $\beta$ -eucryptite sources is limited to 1.5 mA/cm<sup>2</sup> [9]. Emission from zeolites [12] are known to be governed by the rate of surface depletion and diffusing through the material, yielding the relationship jt=constant [13], where j is the current density and t the pulse length. Ueda et al. [14] worked on Li<sup>+</sup> emission properties of pasty, liquid and glass  $\beta$ -eucryptite ion sources, and indicated that there was initially high emission followed by a rapid two-fold reduction of the beam extraction during the first 30 minutes, then a further reduction at a slower rate, down to a quarter of its initial value in 160 min. Another critical issue is that a high temperature ( $\sim 1200^{\circ}$  C) is required to obtain high current Li<sup>+</sup> emission. In comparison, a relatively low temperature of 980 -  $1100^{\circ}$ C is used to extract beams of K<sup>+</sup> and  $Na^+$  or  $Cs^+$  [15] above several mA/cm<sup>2</sup>.

The literature shows that the measured beam current density, from a Li source, is generally not as high as emission of K<sup>+</sup>, Na<sup>+</sup> or Cs<sup>+</sup> [15]. The melting temperature of the lithium alumino silicate is well defined in its phase diagram for  $\beta$ -spodumene and  $\beta$ -eucryptite phases,

but the condition of chemical processing and sintering on large area substrates are not well defined in the literatures. Therefore, we are developing high quality  $\beta$ -spodumene and  $\beta$ -eucryptite Li ion sources for use in the next phase of neutralized drift compression experiments [16]- [17] known as NDCX-II.

#### SOURCE PREPARATION

#### $\beta$ spodumene, $\beta$ eucryptite

Lithium carbonate, aluminum oxide and silicon dioxide have been mixed following stoichiometric ratio in normal atmospheric temperature and pressure in order to make  $\beta$ spodumene of  $Li_2O - Al_2O_3 - 4SiO_2$ . This mixture has been heat treated on a platinum crucible in an air furnace at 800<sup>0</sup>C for two hours to emit carbon dioxide. In the second phase of heat treating, the mixture was re-ground, reheated in the furnace at 1300°C for two hours for further out gassing and to form a spodumene phase. The reacted aluminosilicate has a white or light gray appearance. After separation from the crucible, the substance has been ground with a mortar and pestle until it will pass through a 200  $(75\mu m)$ , 270  $(53\mu m)$  and 400  $(\leq 37 \mu m)$  mesh sieves. In the case of  $\beta$ -eucriptite the stoichiometric ratio is followed as  $Li_2O - Al_2O_3 - 2SiO_2$ . The mixture has been heat treated at 1450° C for 15 minutes to exhaust CO<sub>2</sub> and the outcome is shown in Fig. 1.



Figure 1:  $\beta$ -eucryptite after baking at 1450 °C for 15 minutes, this is followed by grinding with a mortar and pestle until it passes through 200, 270 and 400 mesh sieves.

#### Substrate preparation and coating

A porous tungsten substrate, work function of 4.32-5.22 eV, with 80% density was clean-fired at  $1800^{\circ}$ C to elimi-

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nate any contamination. 400-mesh, Li  $\beta$  spodumene powder was mixed with de-ionized water in a glass beaker and prepared in a very dilute aqueous slurry. Two droplets of aqueous slurry suspension were applied on the substrate and allowed (12 hours) to dry slowly. Next, 200 mesh and 270 mesh particles were mixed into a slurry with de-ionized water and applied on the first layer to make a thicker layer (0.37 mm). The coated substrate was transferred to a  $\geq$ 90% humidity control chamber for the drying process. The humidity was gradually reduced to 6% humidity in 20% decrement steps every 12 hours.

#### Sintering of the coated substrate

The coated substrate was sintered in an argon (with 4% hydrogen) filled oven at  $10^{-2}$  Torr. The temperature of the oven was increased to  $400^{0}$ C in one hour. Then the oven ramped up to  $1430^{0}$ C with a 5 minute flat top at the sintering temperature. The temperature increase and decrease rates were  $10^{0}$ C/m. These are shown in Fig. 2.

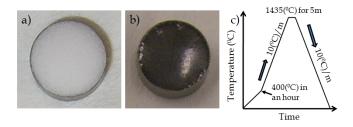


Figure 2: (a) 80% density tungsten substrate, area of  $0.317 \text{cm}^2$ , was coated with  $\beta$ -spodumene, dried in a humidity control chamber for a period of three days; (b)  $\beta$ -Spodumene sintered on the tungsten surface, sintered in a vacuum furnace with 96% Ar with 4% H<sub>2</sub> and at pressure of  $4.5 \times 10^{-2}$  Torr and; (c) Temperature cycle of the sintering process of (b).

#### THE INJECTOR AND SETUP

In the injector, a Li  $\beta$ -spodumine source with tungsten based substrate was installed with a 60 degree Pierce electrode. The gap between the Pierce electrode and the emitter surface was 0.4 mm, a source of some nonlinear aberrations. The extraction electrode aperture has a 1-cm diameter aperture (ground potential). The distance between the source surface and the mid-plane of the extraction electrode is 1.48 cm. The beam diagnostics column is less than a half meter long, accompanied with a 0.1 mm slit, E x B Wein filter, scintillator, and a Faraday cup.

Fig. 3 shows the injector geometry and ion beam trajectories calculated using the WARP particle-in-cell simulation code. A 20 kV extraction voltage predicts a beam current of 8.5 mA of Li from a source of cross sectional area 0.317cm<sup>2</sup>. The single particle transit time inside the injector is 55 ns.

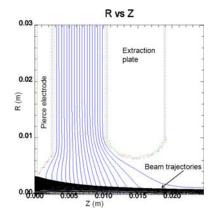


Figure 3: Equipotential lines and beam trajectories of the ion source test injector using the WARP code.

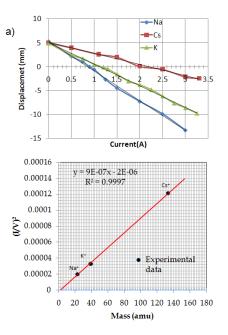


Figure 4: (a) Displacement of Na<sup>+</sup>, K<sup>+</sup> and Cs<sup>+</sup> calibration beams by the E x B filter, operated. The distance from the center of the ExB to the scintillator is  $\sim$ 27 cm, A total votage of 200V with the electrostatic deflection plates was used; (b) calibrated line to determine the elements.

#### DIAGNOSTICS

Verification of ion species, such as Li is especially important. The Li beam could be contaminated due to the source preparation process. Two types of diagnostics were chosen. One of them was the ExB filter, consisted of a dipole magnet and a pair of electrostatic deflection plates. The unknown mass/charge ratio may be determined by:

$$m = 2qV \left(\frac{B}{E}\right)^2 \tag{1}$$

Where, m is the ion mass, q is the ion charge, qV is the ion kinetic energy, B is the magnetic field, and E is the electric field between the two deflection plates. Figure 4 shows

the calibration of the filter with three known sources. Figure 5 shows an example of the identification of  $K^+$  using the filter. The problem with the combination of the aper-

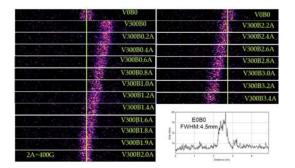


Figure 5: Use of the ExB filter to identify  $K^+$  from a  $\beta$ -spodumine K source.

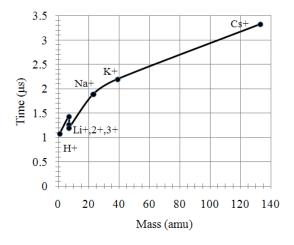


Figure 6: Calculated beam head arrival time of different elements at the scintillator signal location using time of flight standard method. The distance of path length for time of flight measurement is 41.66 cm. A 19.75 kV extraction voltage is used, and a delay of 0.865  $\mu$ s is considered.

ture beam, short pulsed beam and the E x B filter has been that the beam intensity should be high enough to be detected by the scintillator and the gated CCD camera. Low signal-to-noise may ultimately prevent clear identification of low-level contamination of unwanted ion species, The other method is the well known time of flight measurement. Figure 6 shows calculated beam head arrival time of several elements for the present setup.

#### RESULT

Figure 7 shows a pulsed beam signal (yellow line) from a  $\beta$ -Spodumene Li test source, operated at T <1200<sup>0</sup>C. In this initial stage, three species are identified as Li<sup>+</sup>, K<sup>+</sup> and Cs<sup>+</sup>. K<sup>+</sup> and Cs<sup>+</sup> may be expected as contaminates since we used the facility with these elements before. Possible vacuum system contamination will be verified in upcoming measurements. We will also upgrade the source heater

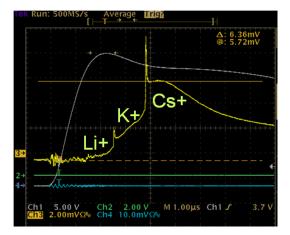


Figure 7: Beam signal (yellow line) showing  $Li^+$ ,  $K^+$  and  $Cs^+$ ; 19.74 kV extraction voltage (white line).

to enable emitter surface temperatures exceeding 1200<sup>0</sup>C.  $\beta$ -spodumene and  $\beta$  eucryptite Li sources to be tested to quantify current density.

Due to the observed significant fraction of impurity and low Li current density, improvement is needed to obtain useful data using the EXB filter. Elimination of contamination and operating with high temperature are in progress.

#### REFERENCES

- J. Barnard et al., Nucl Instrum. and Methods in Physics Research A 577,(2007) 275.
- [2] D. H. McNeill et al, Bull. Am. Phys. Soc. 27, (1982) 1135.
- [3] J. P. Blewett et al., Phy. Review, 50, (1936) 464.
- [4] S. K. Allison et al., Rev. Sci. Intrum., 32 (1961) 1090.
- [5] R. K. Feeney et al., Rev. Sci. Intrum., 47 (1976) 964
- [6] Thomas et al., Rev. Sci. Instrum., 59 (1988), 1735.
- [7] D. M. Thomas, Rev. Sci. Intrum., 66 (1995) 806.
- [8] F. M. Johnson, RCA Rev. 23, (1962) 427.
- [9] A Septier et al., Nucl. Instrum. Methods, 29(1964) 257.
- [10] C. B. Wharton et al., proc. IEEE int. Cons. P. Sci., IEEE 801544-6, (IEEE, New York, 1980)p37.
- [11] Y. Zhang et al., Rev. Sci. Instrum., 78 (2007) 013302.
- [12] A. N. Pargellis et al., J. Appl. Phys. 4999), (1978) 4933-4938
- [13] J. Matossian et al., Bull. Am. Phys., Soc. 24(1979) 1000.
- [14] M. Ueda et al, J. Phys.D: Appl. Phys. 30 (1997) 2711-2716].
- [15] B. Baca et al., PAC05 proceeding, 2005.
- [16] P. A. Seidl et al., these proceedings.
- [17] F. M. Bieniosek, Nucl. Instrum. Method. Phy. Res. A, to be published (2009).