



# Spreading of lithium on a stainless steel surface at room temperature



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## ABSTRACT

Lithium conditioned plasma facing surfaces have lowered recycling and enhanced plasma performance on many fusion devices and liquid lithium plasma facing components are under consideration for future machines. A key factor in the performance of liquid lithium components is the wetting by lithium of its container. We have observed the surface spreading of lithium from a mm-scale particle to adjacent stainless steel surfaces using a scanning Auger microprobe that has elemental discrimination. The spreading of lithium occurred at room temperature (when lithium is a solid) from one location at a speed of 0.62  $\mu\text{m}/\text{day}$  under ultrahigh vacuum conditions. Separate experiments using temperature programmed desorption (TPD) investigated bonding energetics between monolayer-scale films of lithium and stainless steel. While multilayer lithium desorption from stainless steel begins to occur just above 500 K ( $E_{\text{des}} = 1.54$  eV), sub-monolayer Li desorption occurred in a TPD peak at 942 K ( $E_{\text{des}} = 2.52$  eV) indicating more energetically favorable lithium–stainless steel bonding (in the absence of an oxidation layer) than lithium–lithium bonding.

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## 1. Introduction

Liquid plasma facing materials avoid the serious issues with radiation damage, helium blisters, thermal fatigue, and erosion lifetime that affect solid plasma facing components (PFCs) in a fusion reactor. Though less developed than solid plasma facing components, liquid PFCs enable the optimization of a liquid material for high particle flux and heat loads and the independent optimization of the solid, containing material for neutron loading [1]. Liquid lithium has the further advantage of binding with hydrogen isotopes, and lithium conditioning has reduced recycling and enhanced plasma performance on many fusion devices. Lithiumization of carbon plasma-facing components led to substantial advances in plasma performance in TFTR [2]. These were followed by experiments with a liquid Li capillary pore system at T11-M [3] and FTU [4], with a liquid Li tray in CDX-U [5] and with lithiumization of the TJ-II stellarator [6]. Also, a new liquid Li tokamak (LTX) began operation in 2010 [7]. Lithiumization of ATJ graphite plasma facing tiles in the National Spherical Torus Experiment

(NSTX) has shown strong beneficial effects such as improved confinement and reduction and elimination of ELMs [8,9]. A recent overview of lithium applications for fusion devices is given in Ref. [10].

More broadly, the spreading of liquid metals is of high technological importance in areas such as soldering, brazing, and micro-electronic fabrication, however the fundamental surface chemistry of the spreading of reactive liquids is not well established [11]. The contact angle at the liquid/solid interface has traditionally been used as a measure of wettability and interpreted in terms of thermodynamic quantities such as the far-field interfacial energy [12]. Surface energies control which of the three modes of equilibrium film growth occur on surfaces: (i) island or Volmer–Weber, (ii) layer plus island or Stranski–Krastanov, and (iii) layer by layer or Frank–van der Merwe [13]. At the atomic scale, surface energies and film growth modes depend on the relative strength of chemical bonding between atoms of the liquid compared to the bonding strength between atoms of the liquid and the solid substrate.

Capillary wicking of molten lithium on laser-textured 316 L stainless steel and a TZM alloy at 866 K was observed at speeds up to 1.2 cm/s [14]. Lithium films easily react with oxygen and water vapor [15] and the lithium oxide was reported to inhibit wetting of 316 L stainless steel at temperatures up to 723 K [16]. A 1978

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lithium literature review [17] reported “Purified lithium reportedly will not wet stainless steel at 315 °C [588 K] but does at 400 °C [673 K]. Impure lithium will not wet stainless steel at temperatures below 482 °C [755 K].” [18]. The temperature dependence of the contact angle of liquid lithium on stainless steel, Mo, TZM alloy, Ta, and W has been studied and a minimum temperature of 588 K was reported to be necessary for wetting [19]. Cleaning the surface with an argon glow discharge lowered this temperature to 570 K indicating the influence of surface contaminants and topology on the wetting process.

Diffusion of lithium on a Mo (112) single crystal surface was studied by a contact potential technique [20]. Data on the diffusion of Li out of an initial deposit 4.6-monolayers thick showed an extended flattened region at coverages above a monolayer ( $1.1 < \theta < 2.5$ ) that was consistent with an “unrolling carpet” model [21]. A related, detailed investigation of the emission of Sn from Sn islands on an Al substrate was performed using Atomic Force Microscopy (AFM) and scanning Auger microscopy (SAM) [22]. AFM images showed Stranski–Krastanov film growth with Sn islands bounded by crystallographic surfaces. After removal of the surrounding monatomic Sn layer by Ar<sup>+</sup> sputter cleaning, the reforming of this layer by a solid state wetting process was observed by repeating Auger line scans. The rate of spreading of Sn was measured to be 0.2–0.6 μm<sup>2</sup>/s. The spreading was found to be inhibited by impurities such as carbon or oxygen and at well-defined smooth crystallographic interfaces. SAM imaging should also be suitable for lithium spreading studies, and SAM imaging of a partially oxidized lithium surface has been demonstrated [23].

## 2. Lithium – stainless steel chemical bonding

Lithium coatings on a 316 stainless steel sample were studied using temperature programmed desorption (TPD) and Auger electron spectroscopy (AES) in a versatile UHV system containing several complementary surface analysis probes [24] that was operated at a base pressure of  $2 \times 10^{-9}$  torr. The stainless steel sample was cleaned of sulfur and phosphorus impurities using 1-keV Ar<sup>+</sup> ion sputtering, and oxygen and carbon were removed by heating to 1100 K. Some phosphorus contamination remained at the surface as measured by AES (<5 at.%). Oxygen and carbon contamination were less than 10 and 5 at.%, respectively. Lithium was deposited onto the stainless steel sample by thermal evaporation from a SAES Getters alkali metal dispenser [25]. TPD was conducted by resistively heating the sample using a 10 K/s linear temperature ramp and monitoring the desorbed Li with a UTI 100C quadrupole mass spectrometer (QMS) with the ionizer in direct line-of-sight of the sample. Temperature control was implemented using a Eurotherm 3508 PID controller, and the temperature was monitored using a type C thermocouple spot-welded to the back of the sample.

Fig. 1 shows the Li (7 amu) QMS signal as a function of temperature during TPD of lithium films of varying thickness. The release of lithium from SAES Li dispenser is dependent on time and temperature and the temperature is controlled by the amount of current passed through the dispenser. The Li dispenser was operated at 7.1 A for 15 s (curve a), 60 s (curve b), 120 s (curve c), and 180 s (curves d and e). For curves (c) and (d) the sample was pre-flash heated to 550 and 525 K, respectively. After each TPD experiment, the cleaning process was repeated and the next dose of lithium applied. The smallest Li dose produced a sub-monolayer film and had a Li desorption peak at 942 K (corresponding to a desorption activation energy,  $E_{des}$ , of 2.52 eV [26]). With increasing Li dose, additional peaks at 772 K (2.05 eV) and 632 K (1.67 eV) emerged. These rather narrow Li TPD peaks are thought to be due to Li desorption from the thermal decomposition of lithium

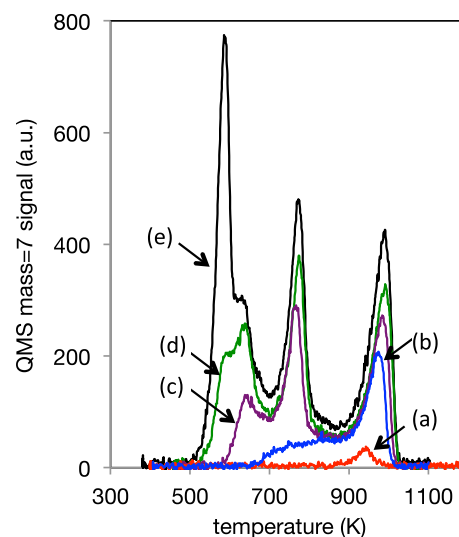


Fig. 1. TPD spectra of lithium (7 amu) desorption measured by a mass spectrometer from lithium films on a stainless substrate. The amount of lithium prior to TPD measurements increases from (a) to (e) – see text.

compounds (e.g. oxides) formed from reaction with contaminants present at the stainless steel surface and/or grain boundaries. After larger Li doses, a low temperature peak appeared, at 585 K for curve (e), which is due to the onset of desorption from a Li multilayer film, where Li–Li bonding controls the energetics. This region of the TPD curve was fit to an Arrhenius expression to give  $E_{des} = 1.54$  eV, which is close to the cohesive energy of metallic Li at 1.69 eV [27].

The desorption activation energy is equal to the adsorption energy in those cases where there is no activation energy of adsorption, as in this case. Thus, for lithium on unoxidized stainless steel, the Li adsorption energy (2.52 eV) is much higher than the lithium–lithium adsorption energy (or binding energy) of 1.54 eV. In the absence of extensive surface oxidation, surface diffusion of lithium over stainless steel is energetically favorable, and a



Fig. 2. Photograph of a particle of lithium pressed onto a 13-mm diameter stainless steel SAM stub.

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