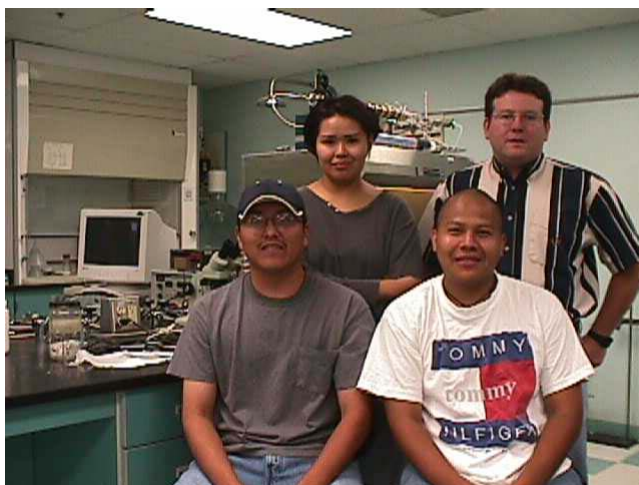


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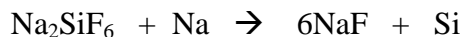
## **Electrodeposition of Silicon using Hexafluorosilicate Salts in Various Solvents**

Summer 2001

Support was from The Alliance for Minority Participation Program, National Science Foundation. The students presented their work at the AMP conference at NMSU September 2001.



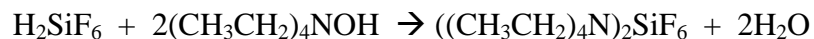
Students Lawendra Atcitty, Chemistry, Derek Chavez (seated left), Engineering, and Mitchell Betonie, Engineering, began the summer's work by making electrical feed throughs for the glove box. They then set out to repeat the experiment of Berzilius using sodium metal, and sodium hexafluorosilicate. The solid materials were placed in a ceramic dish, in the glove box under nitrogen and slowly heated with a heating mantle. At about 150-200C the mixture sparked, then sparked again, and then ignited filling the box with white smoke (probably sodium fluoride). Remains in the dish were a white powder that was soluble in water, and black grey crystals insoluble in water or hydrochloric acid. The mp of the hard black crystals was above 400 C (the limit of our mp apparatus) supporting the production of silicon. Infrared spectroscopy of the black crystals showed the Si-F stretch at 800-1000  $\text{cm}^{-1}$  missing from KBr pellet samples, further indicating elemental silicon. The glove box was carefully scrubbed to remove what we assumed to be sodium fluoride residue (sodium fluoride is highly toxic). The reaction is



The students then proceeded to perform CV scans and potential steps using aluminum working electrode, silicon counter electrode, and platinum reference electrode, of various solutions of sodium hexafluorosilicate, SHFS, ammonium hexafluorosilicate, AHFS, in acetonitrile,

isopropyl alcohol, acetone, also with 15-crown-5 ether additions. All solutions had very low solubility (less than 1% by mass) produced small currents and no discernable silicon deposits.

In an effort to increase the solubility of hexafluorosilicate, Tetraethyl ammonium hexafluorosilicate, TEAHFS was synthesized by metathesis



The TEAHFS crystals were vacuum dried with slight heating to drive off water. The crystals appeared to gel, then melt at 112 C. IR spectra of the compound showed a Si-F stretch at 800-1000  $\text{cm}^{-1}$ , with typical alkane features. The solubility was higher in acetone and isopropyl alcohol again around 10% by mass. Potential step experiments performed on both of these solutions produced thin white deposits. Since no grey black deposits were formed, no further analysis was done on the electrodes.