Investigation of In Situ and Ex Situ Passivation of Pyrophoric Uranium–Niobium Alloy Powder – Supplementary Information

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1. Supplementary Methods

1.1 Sparkle Test Apparatus

Aside from the risk of pyrophoricity, uranium-6 wt.% niobium (U-6Nb) powders pose several other significant safety hazards. U-6Nb metal is radioactive, and the finely divided powder poses a dispersible radioactive material hazard. Additionally, as a heavy metal, finely divided uranium poses a significant toxicity hazard. Because of these additional hazards present even in nonpyrophoric powders, testing apparatus was designed with several engineering controls in place to mitigate the risk of contamination to the laboratory space and personnel.

The testing apparatus was designed to allow for easy loading and testing while reducing the possibility of accidental exposure to air (Figure **S1**). The apparatus consists of a ball valve fitted on one side to a KF blank to store the test sample, with a removable handle affixed to the valve stem. The container is filled with the powder sample inside the glovebox, closed so that the powder remains under argon, and the handle was removed to prevent unintentional opening of the ball valve. It was then moved from the glovebox to a fume hood which contained the custom-build testing apparatus. The ball valve was fixed to a mounting plate positioned above the working surface, and a quarter-circular handle, with cables attached to either end and run-through pullies, was attached to the ball valve. The mounting plate was then lifted to a one meter drop height using a linear actuator. An actuator was used so that the hands and arms of personnel did not at any time need to be underneath the ball valve opening while it was in motion.

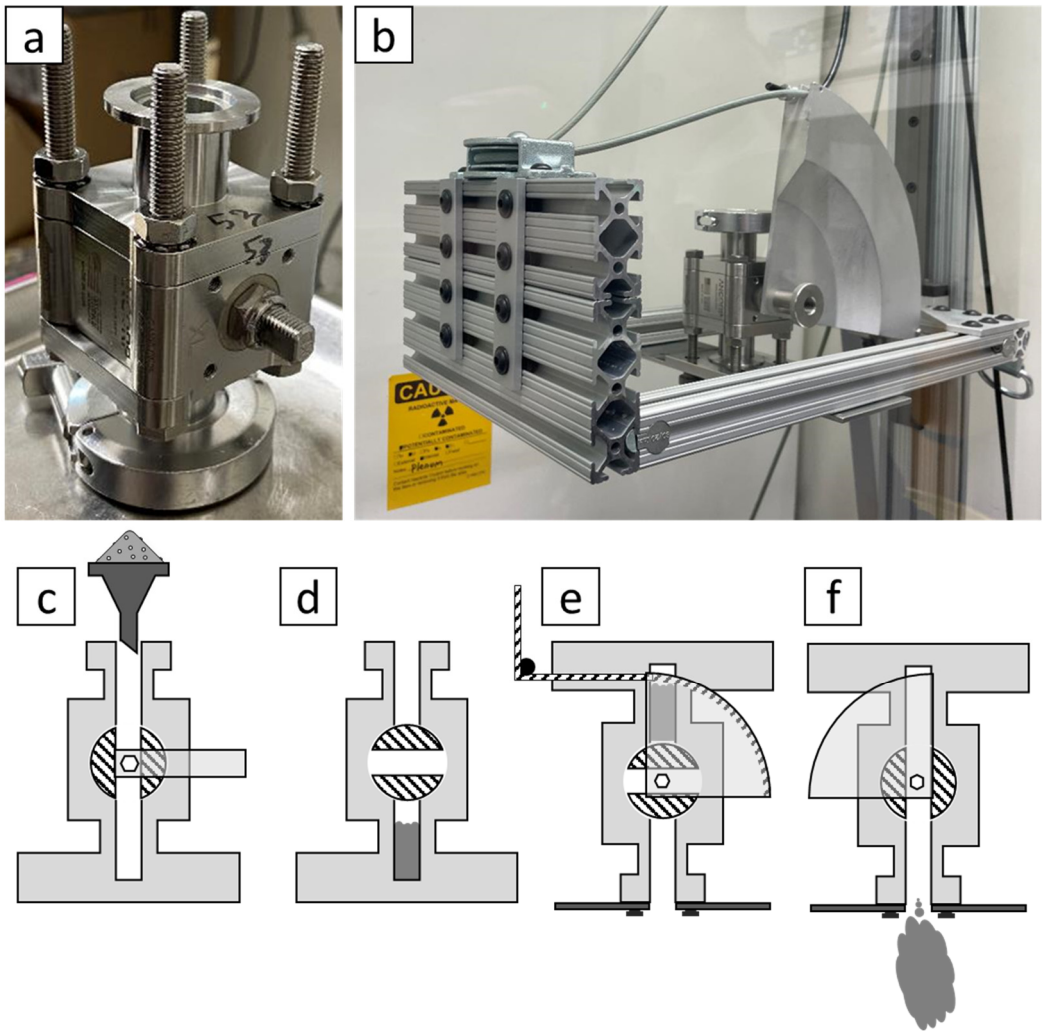


Figure S1: Sparkle test apparatus: (a) ball valve with handle removed and one end shut; (b) ball valve fixed on testing apparatus with pulleys for remote actuation; (c) powder filling container in argon atmosphere; (d) ball valve closed for transportation to testing apparatus; (e) circular quadrant handle fitted to the ball valve with pulleys attached; (f) ball valve opened to drop the powder

After being set to the desired height, a steel catch pan was moved underneath the mounting plate, and a transparent fire-resistant polycarbonate shield was put in place to fully contain any sparks and powder. Finally, the sash of the fume hood was closed, and the test could be performed manually by pulling on each of the two pully cables to open and close the ball valve while standing at a safe distance away from the fume hood. Both the wired and wireless electronic actuation of a butterfly valve was considered. However, to protect against potential connectivity problems in electronic devices, manually operated cables were found to be preferable. The entire apparatus was set up in a well-functioning fume hood, clear of flammable materials, and equipped with a downstream HEPA filter.

This approach allows for robust and repeatable testing for pyrophoricity for a variety of materials with unique and challenging hazards while ensuring the safety of laboratory staff and equipment.

1.2 Sparkle Test

Each powder sample generated was tested for pyrophoricity via the sparkle test. A 1-2 mL aliquot (corresponding to a target mass of approximately 14-16 g) of powder was loaded into a modified manual KF16 ball valve, dropped one meter, and observed for pyrophoric reaction. This test was repeated six times with a new aliquot each time before the powder was deemed non-pyrophoric.

2 Supplementary Results

2.1 Autoignition Point Measurement

When measuring for autoignition temperature following a modified version of ASTM Standard E2021-09, the powder did not ignite until the temperature was artificially raised to 150 ˚C. However, this test only measures a material's autoignition point when undisturbed in a pile in air. When dispersed in air, these powders can generate sufficient heat through exothermic oxidation to ignite without any added heat or spark. In fact, powders which fail the sparkle test can have autoignition temperatures measured through this method of above 150 ˚C. For U-6Nb, the sparkle test was found to be the more sensitive way of determining pyrophoricity.

2.2 Powder X-ray Diffraction (PXRD)

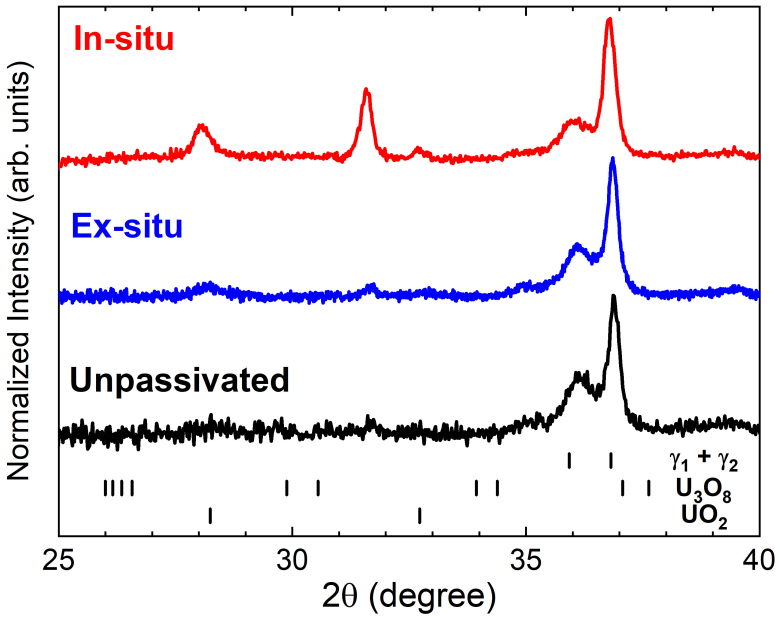


Figure S2: Powder X-ray diffraction of in situ-passivated, ex situ-passivated, and unpassivated U-6Nb powder

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