Statistical Design for Uranium Corrosion Experiments

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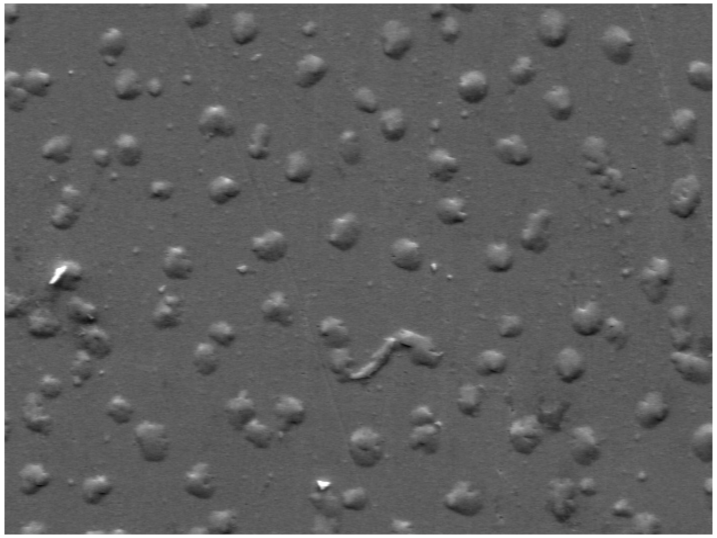
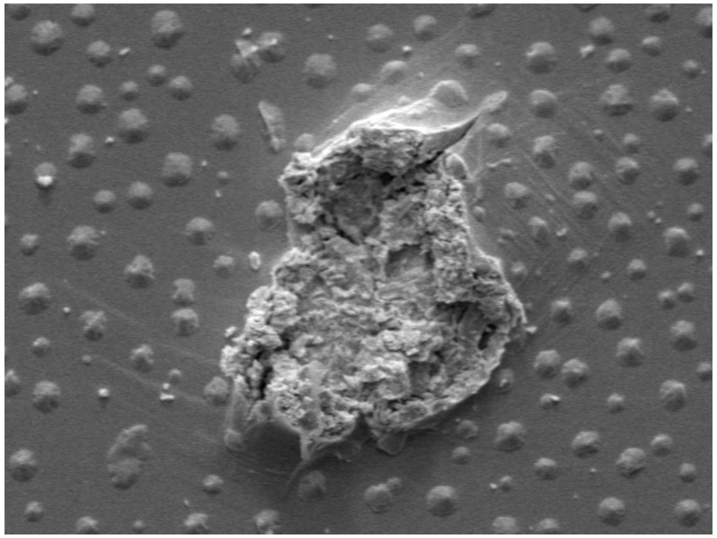
Fig. 1. Blisters on the surface of a uranium coupon sample may be visible early in the uranium corrosion process. Statistical analysis of data from the uranium corrosion study will investigate uranium hydride initiation and growth.

Fig. 2. As the uranium corrosion process continues, the metal surface of the uranium coupon may rupture. A designed experiment provides information about the effects of the different experiment variables.

Statistical experiment design techniques were developed for generating multilevel experiment designs in the presence of a linear trend variable. This work was motivated by the need for an efficient experiment plan for a uranium experiment being conducted by Roland Schulze and Mary Ann Hill, MST-6, to study hydride formation, one of the most important forms of corrosion observed in uranium and uranium alloys. The results of this experiment will contribute to the understanding of uranium corrosion processes. The study goals and objectives of the experiment are described in [1], and the work described here focuses on development of an approach for generating a statistical experiment plan. The experiment planning methods used allow for robust data collection accommodating other sources of variation such as the density of inclusions, which, in this case, varies linearly along the cast rods from which samples are obtained.

The uranium hydride study, now underway, will examine the effects of headspace gas pressure, gap size, and abrasion on uranium hydride initiation and growth. The statistical experiment plan for the uranium hydride study was designed to efficiently collect data to aid in understanding the impact of the experiment factors on initiation and growth of corrosion. Uranium hydride behavior will be measured by quantifying corrosion density, spatial distribution, and volume as a function of aging time on surfaces of uranium coupons. The actual measurements include hydrogen pressure measured over time and digital images, similar to those shown in Figs. 1 and 2, of the metal surfaces from which corrosion can be observed and quantified.





A common statistical approach for experiment planning involves the use of factorial or fractional factorial designs where all or a specially selected subset of all combinations of several variables are included in experimentation [2]. In addition, to collect information on variability, some of the experiment conditions may be repeated. Specifically, for this study, three replicates were planned for each experiment condition. In order to keep the number of possible combinations manageable, generally only two or three values are considered in initial experiment studies. For headspace gas pressure, three values were planned based on results from a preliminary study to investigate reaction rates. Three values for uranium interface gap were selected that included no gap and two different size gaps. The abrasion variable has two values corresponding to either presence or absence of abrasion. Hence, for these three variables, the full factorial experiment set includes 3*3*2 = 18 combinations, so with three replicates each, a total of 54 experiments are planned. Some additional control samples are also included in the experiment.

During sample preparation, the casting procedure for the rods from which the uranium coupons were cut was observed to have variable properties, particularly with respect to inclusions along the length of the rod. Measurements of the density of inclusions in a rod exhibited a clear linear trend. As a result, inclusion density needs to be considered in experiment planning. Since inclusions provide possible hydride corrosion entry points, the number of inclusions is expected to have an effect on the variability in hydride corrosion behavior. Inclusion density effectively adds an additional nuisance variable, along with the three main variables of initial interest for the current experiment plan.

An experiment plan was required that could assign three samples to each of the 18 conditions defined by the three main variables (headspace gas pressure, uranium interface gap, and coupon surface abrasion) such that the anticipated trend with inclusion density does not bias estimation of effects of the three variables of primary interest. This type of experiment planning is referred to in the statistics literature as trend-free experiment design [3-5]. Although the desire for a trend-free design generally arises with an anticipated time trend, and the literature generally considers factorial structure with two or three levels, the new experiment planning issues here include accommodating factorial structure with mixed numbers of levels. The mixed levels structure means that the balance needed to achieve trend-free estimates is not perfectly attainable but only nearly so.

An experiment plan was developed to investigate headspace gas pressure, uranium interface gap, and coupon surface abrasion effects on uranium hydride initiation and growth on coupon samples. The experiment plan was constructed to accommodate the presence of the trend variable associated with density of inclusions varying with location in the cast rods, by carefully assigning samples cut from the different locations on the rod to the combinations of experiment conditions to achieve a nearly trend-free design.

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[1] R.K. Schulze, M.A Hill, LA-UR-08-1879 (2008).
[2] C. Wu, M. Hamada, *Experiments: Planning, Analysis, and Parameter Design Optimization,* John Wiley & Sons, New York (2000).
[3] M.L. Lin, A.M. Dean, *Annals of Statistics* **19**(3), 1582-1596 (1991).
[4] P.W.M. John, *Technometrics* **32**(3), 275-282 (1990).
[5] A.C. Atkinson, A.N. Donev, *Technometrics* **38**(4), 333-341 (1996).

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