PNNL-28495

Pacific Northwest
NATIONAL LABORATORY

Quantifying and Qualifying Alloys Based on Level of Homogenization: A U-10 wt% Mo Alloy Case Study

January 2019

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U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Summary

Homogenization heat treatment is performed to attain uniformity in microstructure of metals and metal alloys, which is helpful to achieve the desired workability and microstructure in final products, and eventually to gain predictable and consistent performance. Fabrication of fuel plates made from a low-enriched uranium alloy with 10 wt% Mo (U-10Mo) involves multiple thermomechanical processing steps. It is well known that the molybdenum homogeneity in the final formed product affects the fuel performance in the nuclear reactor. To make sure these materials are uniformly homogenized, a statistical method was proposed to quantify and characterize the Mo concentration variation in U-10Mo fuel plates by analyzing the Mo concentration measurement data from scanning electron microscopy–energy dispersive spectroscopy line scans. Statistical tolerance intervals were employed to determine the qualification of U-10Mo fuel plates. We formulate an argument for the minimum number of independent samples required to define fuel plate qualification if no Mo measurement data are available in advance, and demonstrate that the given tolerance interval requirements can be equivalently reduced to a sample variance criterion in this application. The outcome of the statistical analysis can be used to optimize casting design and eventually increase productivity and reduce fabrication cost. The statistical strategy developed in this report can be implemented for other applications, especially in the field of material manufacturing to assess qualification requirements and monitor and improve process design.

Acknowledgments

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Acronyms and Abbreviations

ACF	autocorrelation function
CI	confidence interval
EDS	energy dispersive spectroscopy
SE	secondary electron
SEM	scanning electron microscope
TI	tolerance interval
U-10Mo	uranium alloyed with 10 wt% molybdenum

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1.0 Introduction

The U.S. National Nuclear Security Administration drives a need to develop and design low-enriched uranium fuels to progressively reduce and eventually replace high-enriched uranium fuels for U.S. high-power civil and research reactors (Wachs et al. 2008). Uranium alloyed with 10 wt% molybdenum (U-10Mo) has been identified as the most promising candidate for low-enriched uranium fuel, due to its high intrinsic density and good irradiation behavior (Van Den Berghe and Lemoine 2014; Meyer et al. 2002; Snelgrove et al. 1997). The manufacturing of the U-10Mo alloy requires multiple complex thermomechanical processes (Hu et al. 2017; Xu et al. 2016; Soulami et al. 2019; Jana et al. 2019; Nyberg et al. 2019; Prabhakaran et al. 2016) including casting, homogenization, hot/cold rolling, annealing, and hot isostatic pressing. Every process is highly challenging and requires a tremendous amount of planning and preparation. Fuel plate quality after casting and homogenization is fundamental to support high quality of the final U-10Mo product because it can greatly affect the subsequent microstructure evolution and eventually influence the fuel performance in a reactor. To determine the qualification of a given fuel plate, statistical analysis of the chemical composition measured using an energy dispersive spectroscopy (EDS) technique is a reliable, efficient, and cost-effective method. Statistical methods have been widely used to characterize the quality of processing products in many applications (Garrett and Prasad 2004; Ren et al. 2016; Ulewicz 2003; Reid and Sanders 2007). For this project, tolerance interval (TI) calculations will be used to determine the quality of as-cast plates, identify potential quality problems, and eventually enhance the quality and efficiency of the entire fabrication process, thereby reducing fabrication costs.

Previous work (Xu 2016; Wang et al. 2017) has revealed that the distribution of Mo in fuel plates has significant effects on the quality and performance of U-10Mo fuel. In principle, the overall average Mo concentration should be 10% throughout the entire plate. However, the variation of Mo concentration after casting is significant due to Mo segregation, and a homogenization treatment is necessary to produce a more uniform Mo distribution. Therefore, statistical analysis is needed to quantify the variation of Mo concentration that determines the qualification of fuel plates.

Two common measures of product quality compare the confidence interval (CI) for a parameter and/or a TI to specified limits (Fedorov et al. 2014; Rebafka 2007). A CI specifies the statistical interval that might contain the true value of a population parameter, e.g., mean or variance, with a given probability. The width of CI will approach zero with increasing sample size. A TI bounds a specified proportion of a sampled population at a given CI. The width of the TI is also dependent on the number of samples and variance.

Extensive work (Sharma and Mathew 2012) has been performed for quality control in a broad range of applications, where the TI is used as a quantitative metric for assessing the product quality. However, no such attempt has been published on characterizing the Mo concentration variation in U-10Mo fuel plates for fuel qualification. A recent statement of qualification specification for U-10Mo fuel plate has been proposed (INL 2017), and is quoted herein as follows: ***"A sufficient number of samples shall be randomly taken from a sufficient number of fuel plates randomly selected from a fuel plate lot to allow statistical determination of 95% confidence that 95% of the U-10Mo has 10.0 ± 1.0 wt% Mo microscopically throughout the U-10Mo."*** This statement prompts one to use TI to quantify the Mo concentration variation and foster product quality. This specification can be equivalently interpreted as follows: 95% of the total U-10Mo population within a plate must fall between 9 and 11 wt% Mo with a confidence level of 95%. However, there is no guideline on the "sufficient number of samples" in the statement. As in other sampling scenarios, there is a trade-off between the number of high quality measurements that can be obtained and the total time cost for data collection. Specifically, a single Mo wt% measurement from EDS line scans takes about 30 seconds to 1 minute, and a large number of

measurements is required to meet statistical specifications, given inherent Mo variability within a plate. In addition to determining the fuel plate qualification, this study also provides insights into an inverse problem, i.e., how to determine the optimal number of measurements required to qualify the given plate.

We also argue that the fuel plate qualification can be specified in terms of the sample variance, which is a simplified metric that may be applied given reasonable assumptions. One of these assumptions is that the collected samples must be independent of one another, which ensures that the selected data properly represent the characteristics of the entire fuel plate. The product is considered qualified only if the simplified metrics are satisfied and the assumptions can be verified. The final results of the statistical analysis can be used to optimize casting design, refine casting specifications, and eventually enhance casting productivity. The statistical strategy developed in this paper can be implemented in other applications, especially in the field of material manufacturing, to define reasonable qualification requirements and provide insights to improve process design.

The rest of the report is organized as follows. Section 2.0 describes the statistical methodology, including a comprehensive introduction of TI approaches and key mathematical formulas. Section 3.0 presents the TI implementation on the specified U-10Mo qualification specification, and two examples are analyzed as demonstration. Section 4.0 introduces our recommendation on the minimum number of Mo wt% measurements and the sample variance criterion to determine fuel plate qualification. The conclusions are summarized in Section 5.0.

2.0 Methodology

The general definition of a TI can be described as follows. Let $\boldsymbol{Y} = (Y_1, Y_2, ..., Y_N)$ be N independent random samples drawn from a continuous cumulative distribution function, F. In addition, let an interval $[L(\boldsymbol{Y}), U(\boldsymbol{Y})]$ based on the sample vector $\boldsymbol{Y}$ be a two-sided TI for F such that for, any given $0 < \gamma < 1$ and $0 < P < 1$, the following probability expression is satisfied:

$$\Pr(F(U(\boldsymbol{Y})) - F(L(\boldsymbol{Y})) \geq P) = \gamma, \tag{1}$$

where γ and P define a general two-sided TI that has a confidence level of $\gamma \times 100\%$ containing at least $P \times 100\%$ of the population. If $L(\boldsymbol{Y}) = -\infty$ and $U(\boldsymbol{Y}) < +\infty$, then the previous two-sided interval becomes $[-\infty, U(\boldsymbol{Y})]$ and can be defined more properly as a one-sided TI (γ, P) with an upper tolerance bound. Similarly, if $L(\boldsymbol{Y}) > -\infty$ and $U(\boldsymbol{Y}) = +\infty$, the interval $[L(\boldsymbol{Y}), +\infty]$ is a one-sided TI (γ, P) with a lower tolerance bound only (Guenther 1972).

Now, suppose that sample vector $\boldsymbol{Y} = (Y_1, Y_2, ..., Y_N)$ is independently drawn from a standard normal distribution $N(\mu, \sigma^2)$ with a population mean μ and a population variance σ^2. We can use $\overline{Y}$ and s to denote sample mean and sample variance, respectively. The mathematical expressions for $\overline{Y}$ and s can be written as

$$\overline{Y} = \frac{\sum_{i=1}^{N} Y_i}{N}, \tag{2}$$

$$s = \sqrt{\frac{\sum_{i=1}^{N}(Y_i - \bar{Y})^2}{N-1}}\,. \tag{3}$$

$$L(\boldsymbol{Y}) = \bar{Y} - k_1(N, \gamma, P)s \tag{4}$$

or

$$U(\boldsymbol{Y}) = \bar{Y} + k_1(N, \gamma, P)s\,, \tag{5}$$

where k_1 is the factor to ensure that at least a proportion $P \times 100\%$ of the entire population, with confidence $\gamma \times 100\%$, is satisfied based on the sample of size N. The value of k_1 can be calculated based on the inverse cumulative distribution function for a noncentral t distribution (Natrella 2012),

$$k_1(N, \gamma, P) = \frac{t_{N-1,\gamma,\delta}}{\sqrt{N}} \tag{6}$$

where $t_{N-1,\gamma,\delta}$ is the critical value ($\gamma \times 100$ percent), i.e., $F\left(t_{N-1,\gamma,\delta}\right) = \gamma$, for a noncentral t distribution with $N - 1$ degrees of freedom and noncentrality parameter $\delta = z_p\sqrt{N}$. Here, z_P is the critical value of the standard normal distribution associated with a cumulative probability of $P \times 100\%$. For example, Figure 1 (Bognar n.d) shows that the accumulated probability for $-\infty \le x \le 1$ is 84%, which is equivalent to $\Pr[x \le z_p = 1] = 84\%$. The value of Z_P can be found in (Loucks and van Beek 2017) for any given P. Some commonly used P and associated Z_P values are listed in Table 1.

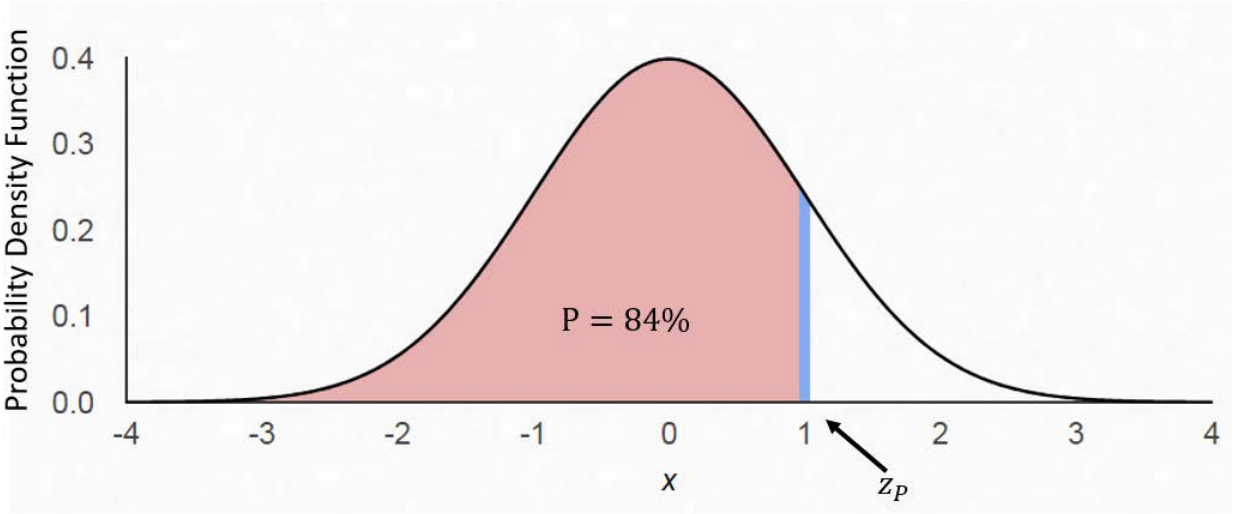


Figure 1. The Critical Value Z_P for a Standard Normal Distribution

Table 1. Critical Values Z_P for Some Commonly Used Probability P Values

P	0.01	0.025	0.05	0.1	0.9	0.95	0.975	0.99
z_P	−2.326	−1.960	−1.645	−1.282	1.282	1.645	1.960	2.326

If N is greater than 50 (Lieberman 1957; Natrella 2013), k_1 can be approximated using the equation below:

$$k_1(N,\gamma,P)=\frac{z_P+\sqrt{z_P^2-ab}}{a} \tag{7-1}$$

$$a=1-\frac{z_\gamma^2}{2(N-1)} \tag{7-2}$$

$$b=z_P^2-\frac{z_\gamma^2}{N} \tag{7-3}$$

Next, the upper and lower bounds for a two-sided TI are defined as

$$L(\boldsymbol{Y})=\bar{Y}-k_2(N,\gamma,P)s \tag{8-1}$$

and

$$U(\boldsymbol{Y})=\bar{Y}+k_2(N,\gamma,P)s \tag{8-2}$$

The exact solution for the two-sided k_2 factor can be obtained by solving the following implicit nonlinear integral equation (Witkovský n.d.; Janiga and Garaj n.d.; Young 2016):

$$\sqrt{\frac{2N}{\pi}}\int_0^{+\infty}\Pr\left(\chi_{N-1}^2>\frac{(N-1)\chi_{1,P,\delta}^2}{k_2(N,\gamma,P)^2}\right)e^{-\frac{1}{2}Nz^2}dz=\gamma\,, \tag{9}$$

where χ_{N-1}^2 is the chi-squared probability distribution function with $N-1$ degrees of freedom. $\chi_{1,P,\delta}^2$ is the $P\times 100\%$ percent of the chi-squared distribution with one degree of freedom and noncentrality $\delta=z^2$, where z is a dummy variable used for integration. Equation (9) is quite complicated to solve directly, while the following approximation can be used to simplify the calculation of the two-sided k_2 factor (Howe 1969:

$$k_2(N,\gamma,P)=\left(\frac{(N-1)\left(1+\frac{1}{N}\right)z_{\frac{1+P}{2}}^2}{\chi_{1-\gamma,N-1,0}^2}\right)^{1/2}\,, \tag{10}$$

where $\chi_{1-\gamma,N-1,0}^2$ is the critical value ($1-\gamma$ percent) of the central chi-squared distribution with $N-1$ degrees of freedom and noncentrality parameter $\delta=0$, i.e., $F\left(\chi_{1-\gamma,N-1,0}^2\right)=1-\gamma$. Figure 2 shows an

example chi-squared distribution with $N - 1 = 3$. The probability of having a value greater than $\chi^2_{1-0.57,3,0} = 2$ is 57%, which is equivalent to $\Pr[x \geq \chi^2_{1-\gamma,N-1,0} = 2] = 57\%$. The values of $\chi^2_{1-\gamma,N-1,0}$ for any given γ and N can be found in (Loucks and van Beek 2017). Some commonly used critical values of chi-square distribution are listed in Table 2.

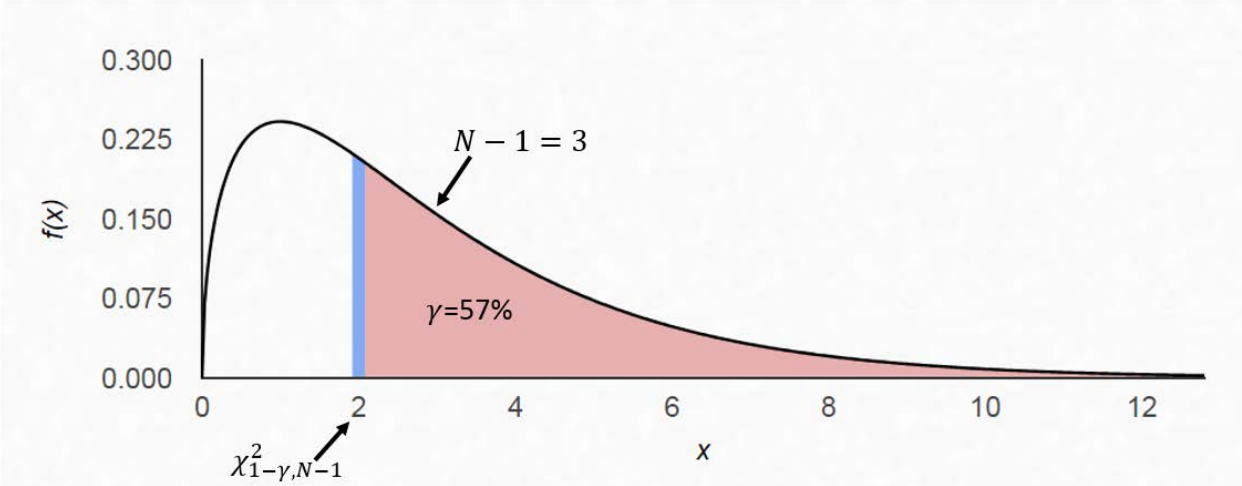


Figure 2. The Critical Value for a Central Chi-Squared Distribution with $\boldsymbol{N-1=3}$ and $\gamma = 57\%$

Table 2. Commonly Used Critical Values ($\chi^2_{1-\gamma,N-1,0}$) of Chi-Square Distribution with Different $1-\gamma$ and $N-1$

N − 1	**1 − γ**				
	0.1	**0.05**	**0.025**	**0.01**	**0.001**
1	0.016	0.004	0.001	0	0
2	0.211	0.103	0.051	0.02	0.002
3	0.584	0.352	0.216	0.115	0.024
4	1.064	0.711	0.484	0.297	0.091
5	1.61	1.145	0.831	0.554	0.21
6	2.204	1.635	1.237	0.872	0.381
7	2.833	2.167	1.69	1.239	0.598
8	3.49	2.733	2.18	1.646	0.857
9	4.168	3.325	2.7	2.088	1.152
10	4.865	3.94	3.247	2.558	1.479
15	8.547	7.261	6.262	5.229	3.483
20	12.443	10.851	9.591	8.26	5.921
25	16.473	14.611	13.12	11.524	8.649
30	20.599	18.493	16.791	14.953	11.588
35	24.797	22.465	20.569	18.509	14.688
40	29.051	26.509	24.433	22.164	17.916
45	33.35	30.612	28.366	25.901	21.251

N − 1	$1-\gamma$				
	0.1	**0.05**	**0.025**	**0.01**	**0.001**
50	37.689	34.764	32.357	29.707	24.674
55	42.06	38.958	36.398	33.57	28.173
60	46.459	43.188	40.482	37.485	31.738
65	50.883	47.45	44.603	41.444	35.362
70	55.329	51.739	48.758	45.442	39.036
75	59.795	56.054	52.942	49.475	42.757
80	64.278	60.391	57.153	53.54	46.52
85	68.777	64.749	61.389	57.634	50.32
90	73.291	69.126	65.647	61.754	54.155
95	77.818	73.52	69.925	65.898	58.022
100	82.358	77.929	74.222	70.065	61.918

To evaluate the accuracy of the k_2 approximation from Eq. (10), a comparison is conducted by solving the true solutions calculated from Eq. (9) using the R package developed in (Young 2016). The relative error between approximation and true solution is defined as $1 - \frac{k_2(approximation)}{k_2(true\ solution)}$. Figure 3 plots the number of samples N versus the relative error for $P = \gamma = 0.95$. The approximations first slightly underpredict and then overpredict the true value of k_2 with increasing N, but the relative error is always within the range of ±0.36% and gradually approaches zero with increasing N. These results indicate that the k_2 values obtained from Eq. (10) can be considered as good approximations to represent the true solutions for N approaching 100.

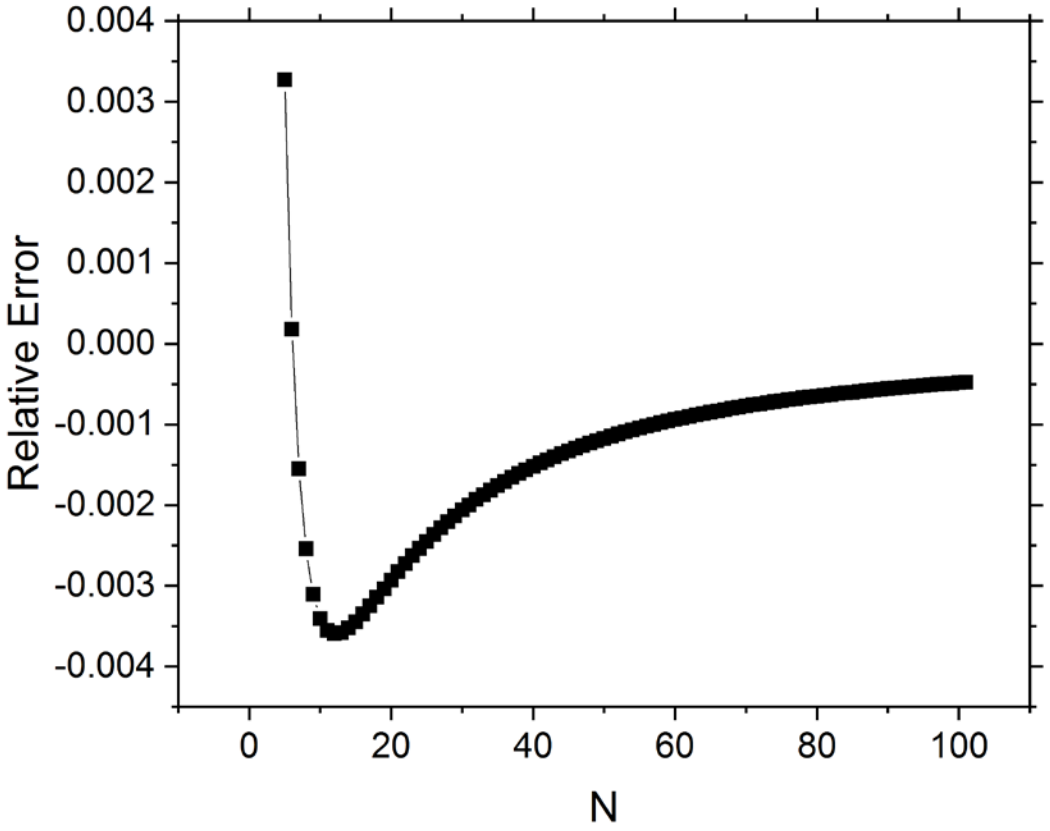


Figure 3. Relation Between Relative Error and N for k_2

3.0 Statistical Analysis for Qualification of U-10Mo

Based on the specification statement for U-10Mo, we can set $P = \gamma = 0.95$. In addition, this requirement also defines the lower and upper bounds for 95/95 TI as 9% and 11%, respectively. Note that an average concentration $\overline{Y} = 10\%$ is assumed for all samples, and is assumed known without error so Eqs (4), (5), and (8) become functions of k_2 and s. The number of samples, N—namely, the number of measurements of Mo concentration required when performing EDS scan experiments for each fuel plate—is not explicitly specified and will be determined in this study. Mo concentration will drop significantly where the scan intersects a carbide or secondary-phase area. To avoid the bias due to carbides, only Mo weight percentages between 7% and 13% are considered effective measurements and used in the plate qualification analysis. The effect of distribution of carbides and/or secondary-phase areas on the fuel plate quality is currently not considered in this study.

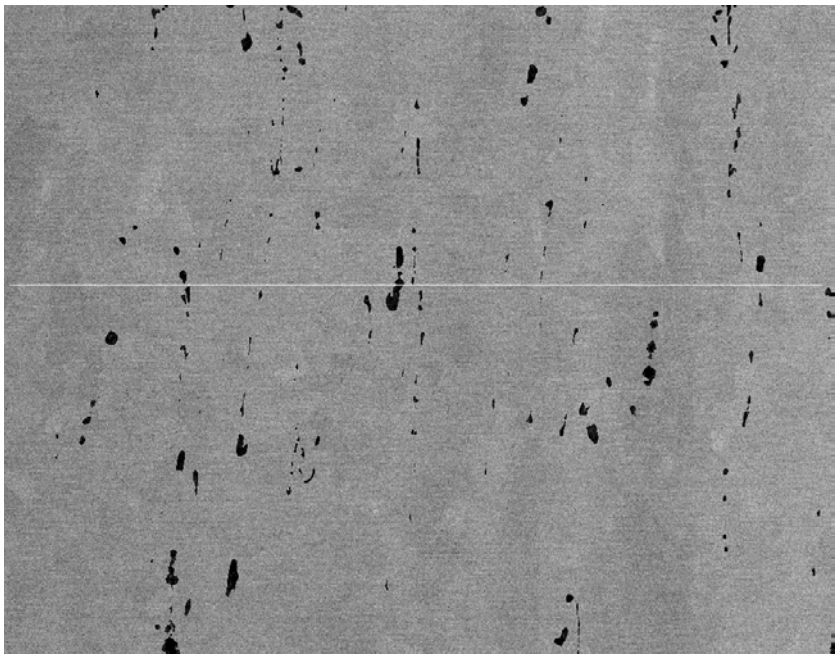
For any independently drawn Mo concentration vector of length N that is in the neighborhood of 100, Eq (10) can be applied to calculate the k_2 factor. With $P = 0.95$, we can find $z_{(1+P)/2} = z_{0.975} = 1.96$. The values of $\chi^2_{1-\gamma,N-1,0} = \chi^2_{0.05,N-1,0}$ and $k_2(N,\gamma,P) = k_2(N,0.95,0.95)$ become functions of N only and are calculated once N is provided. Equation (8) can be used to determine lower and upper bounds (L and U) for the observed vector of Mo concentrations. The fuel plate is determined to be qualified in terms of the Mo distribution if both the upper and lower bounds satisfy the specification requirements, i.e., $L \geq 9\%$ and $U \leq 11\%$. Otherwise, the plate will be rejected.

Two examples with EDS line-scan measurements of Mo concentration were used to demonstrate the qualification determination procedure. The secondary electron (SE) image from the scanning electron microscope (SEM), together with its EDS line scan for the first sample fuel plate (Plate 1) is shown in Figure 4. Figure 5 shows the observed probability density function from a total of 999 Mo concentration measurements taken along the line. Figure 5 shows a bimodal distribution with two peaks evident, the smaller of which represents the carbide regions. To exclude the anomalies from the carbides, only 946 observations are considered effective measurements, with Mo concentrations between 7 and 13 wt%. Given the exclusion of carbides, Mo wt% is expected to be normally distributed and consistent with the assumptions described in Section 2.0.

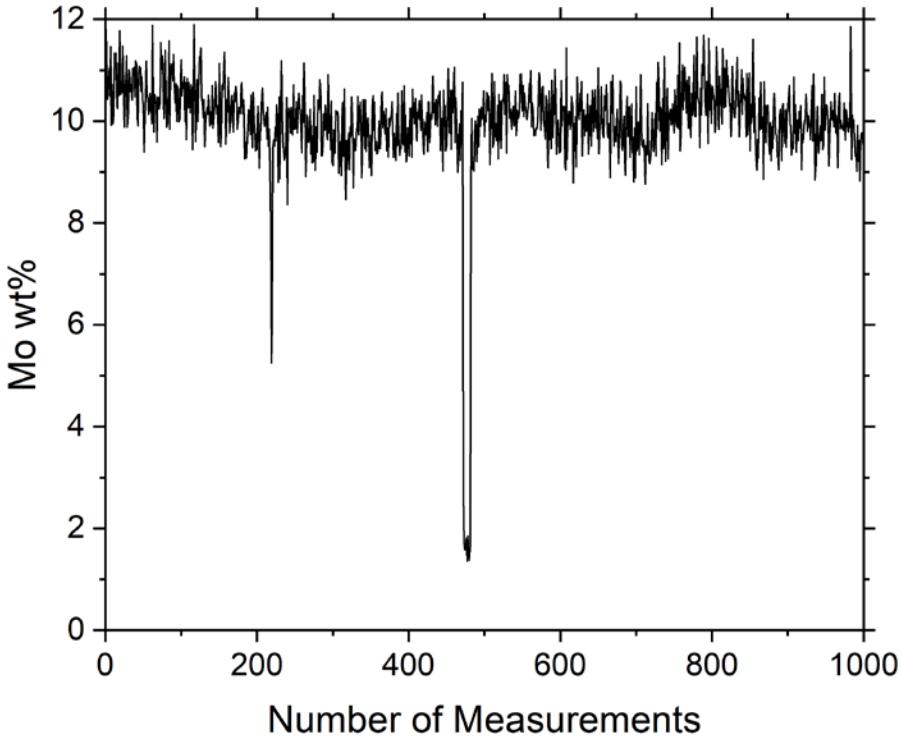
Next, the interdependence of the Mo concentration measurements for Plate 1 is explored using the autocorrelation function (ACF), which is a commonly used method for detecting nonrandomness in data. Given equally spaced measurements, the autocorrelation function r for lag j is defined as

$$r_j = \frac{\sum_{i=1}^{N-j}\left(Y_i - \bar{Y}\right)\left(Y_{i+j} - \bar{Y}\right)}{\sum_{i=1}^{N}\left(Y_i - \bar{Y}\right)^2}. \tag{11}$$

A unit lag distance (j = 1) in our study is 1 μm, which represents the space between the adjacent Mo concentration measurements. By observing the line-scan data listed in Figure 4(b), it is evident that the measurements follow a sinusoidal function, which is a strong indication of data correlation. This trend becomes even more obvious if carbides are excluded. The findings from Figure 6, which plots the ACF results for Plate 1, are consistent with the observations from the measured data. In particular, the critical lag distance indicating no data intercorrelation increases from ~15 μm to ~100 μm after neglecting carbides. The critical lag distance is approximated at the place most of the ACF values start to drop below the top blue dashed line in Figure 6, which specifies a 95% CI of data independence.



(a)



(b)

Figure 4. SE Image (a) Where the White Line Indicates the Position of the Line Scan**,** and EDS Line Scan (b) for Plate 1

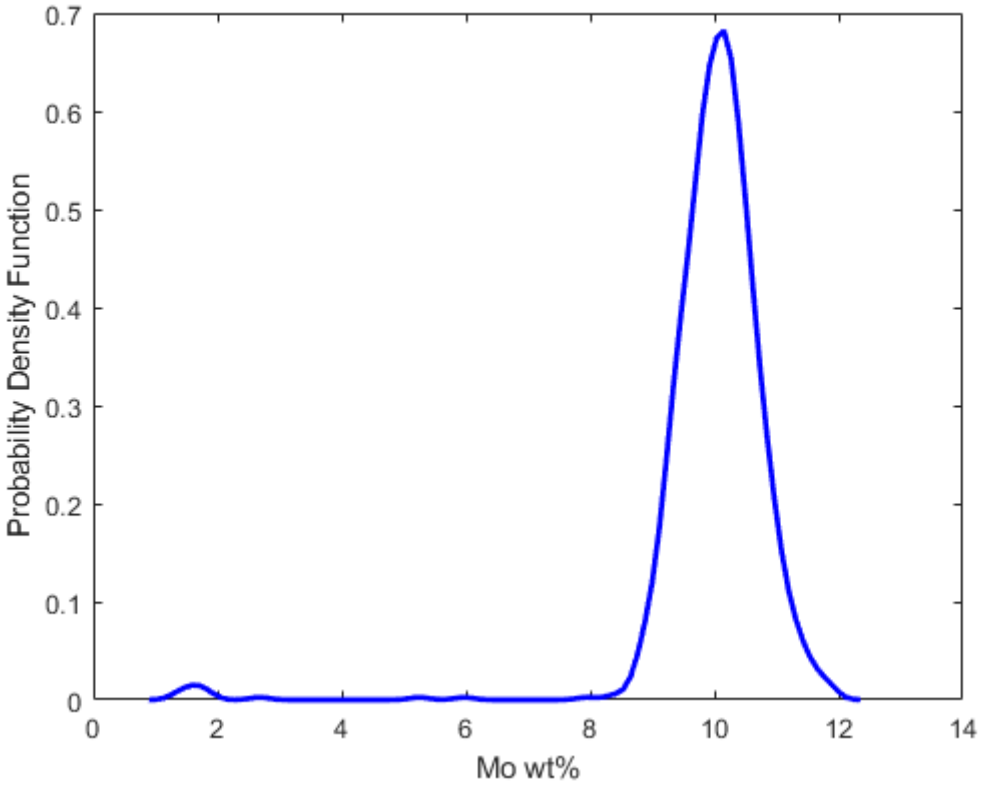
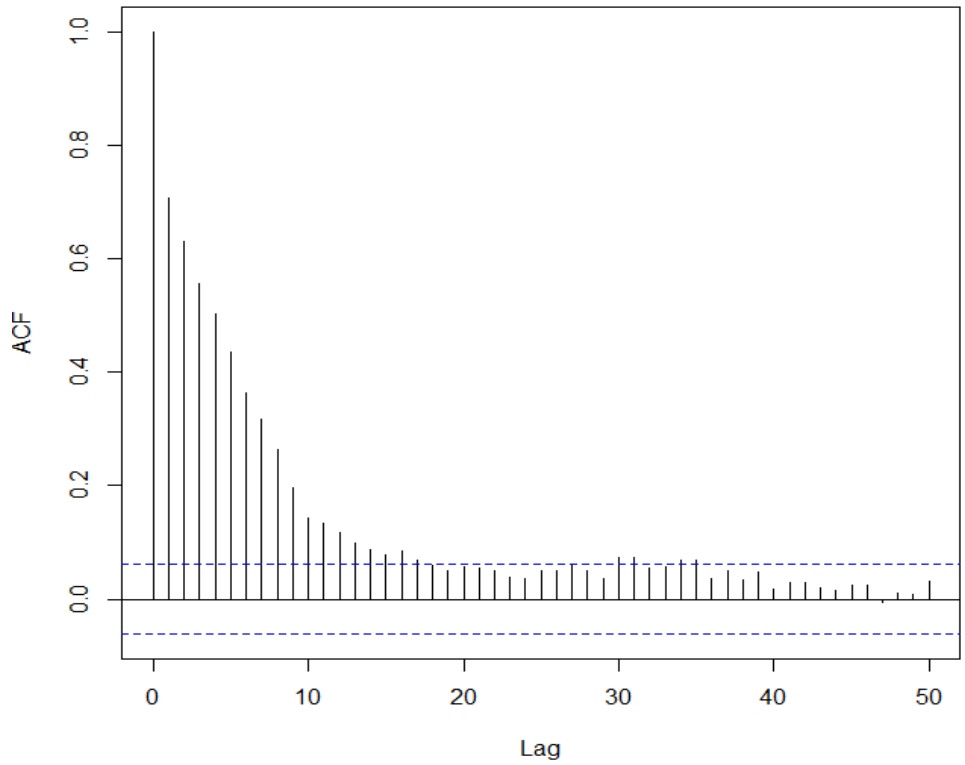
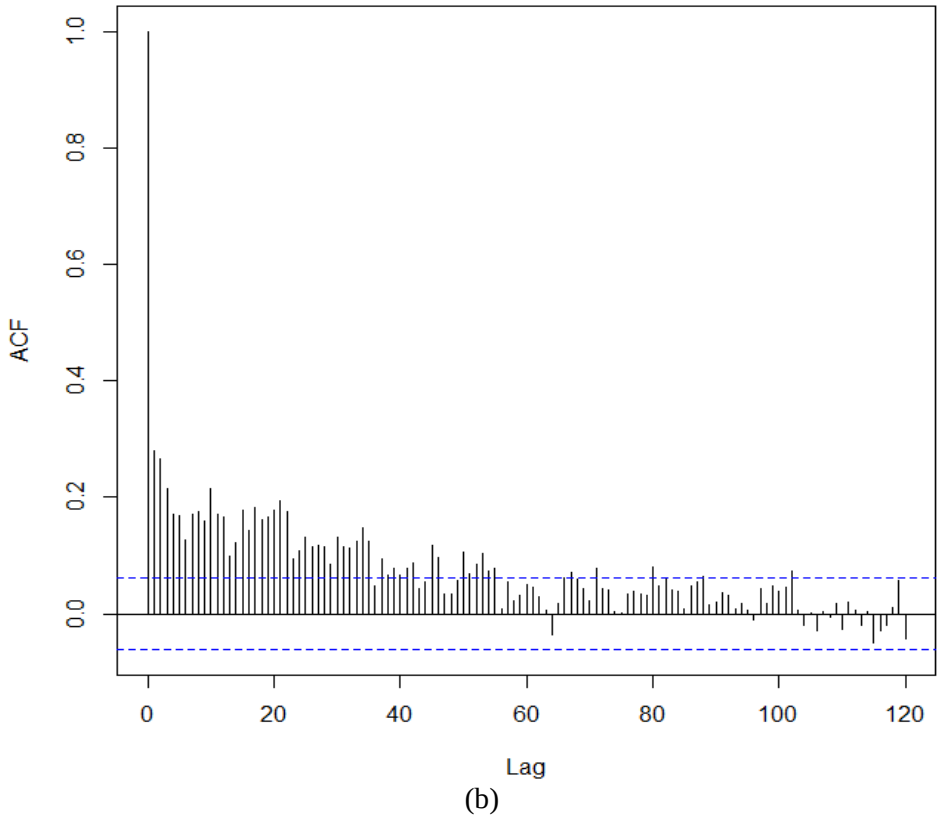


Figure 5. Probability Density Function of 999 Mo wt% Measurements



(a)



(b)

Figure 6. ACF Results for Plate1 with (a) and without Particles (b)

To simulate the distribution of k_2 as a function of the number of samples, we randomly sampled N_s samples from the observed data vector, repeating the experiment 1000 times for each value of N_s in Table 3. The last column in this table shows the results for N_s = N =946. For any given N_s the k_2 for 95/95 TI can be calculated from Eq. (10) and appear in the third row of Table 3. The mean value of sample standard deviation, s, listed in the fourth row slightly increases with N_s and then remains constant for N_s greater than 20.

The relationship between sample variance and population variance is expressed as Kenney and Keeping (1951)

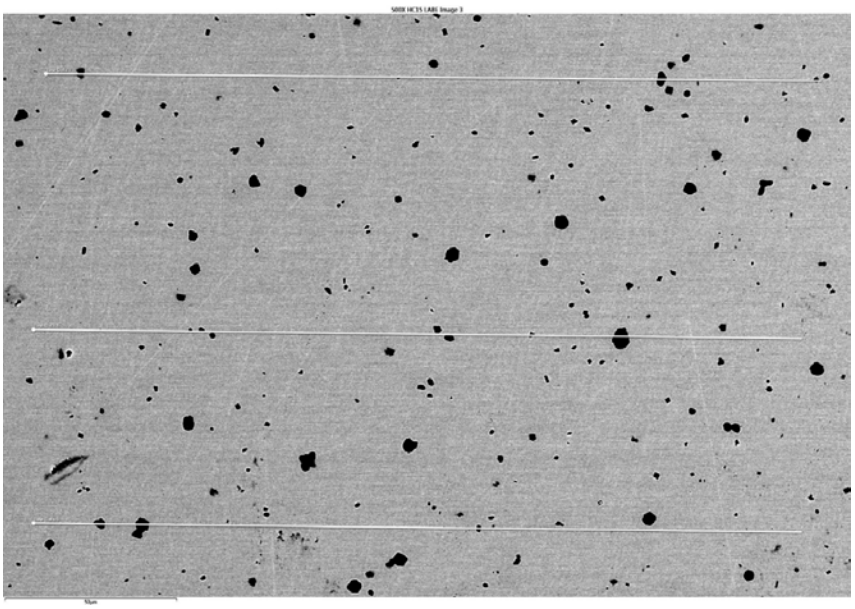
$$s^2 = \left(1 - \frac{1}{N}\right)\sigma^2. \tag{12}$$

Equation (12) shows that sample variance will gradually increase and approach the population variance (constant) with increasing N, which is consistent with our findings. The next row of Table 3 shows the mean value of $k_2 s$ (multiplication of k_2 and s), which decreases with increasing sample size N_s. The last two rows list the corresponding lower and upper bounds for each N_s. $L < 9\%$ and $U > 11\%$ are observed for all cases, which indicates that this plate will not meet specified qualification because the Mo spatial distribution is not sufficiently homogeneous.

Table 3. Effect of Number of Samples on Plate Qualification for Plate 1

N_s	**5**	**10**	**20**	**50**	**100**	**200**	**946**
Repetition	1000	**1000**	1000	1000	1000	1000	1
k_2	5.09	**3.38**	2.75	2.37	2.23	2.14	2.03
Mean (s)	0.53	**0.55**	0.56	0.57	0.57	0.57	0.57
Mean ($k_2 s$)	2.58	**1.77**	1.45	1.30	1.21	1.18	1.12
Lower Bound (L)	7.42	**8.23**	8.55	8.70	8.79	8.82	8.88
Upper Bound (U)	12.58	**11.77**	11.45	11.30	11.21	11.18	11.12

Next, a second example plate (Plate 2) with its SEM and three EDS lines (250 measurements on each line) is shown in Figure 7. The statistics for varying N_s on plate qualification for Plate 2 are listed in Table 4. The minimum value of N_s to make $L > 9\%$ and $U < 11\%$ and reach qualification requirement is 10 for Plate 2. The corresponding column is highlighted in bold in Table 4. Therefore, the Mo distribution in Plate 2 is determined to be sufficiently homogeneous to qualify for the subsequent fabrication processes.



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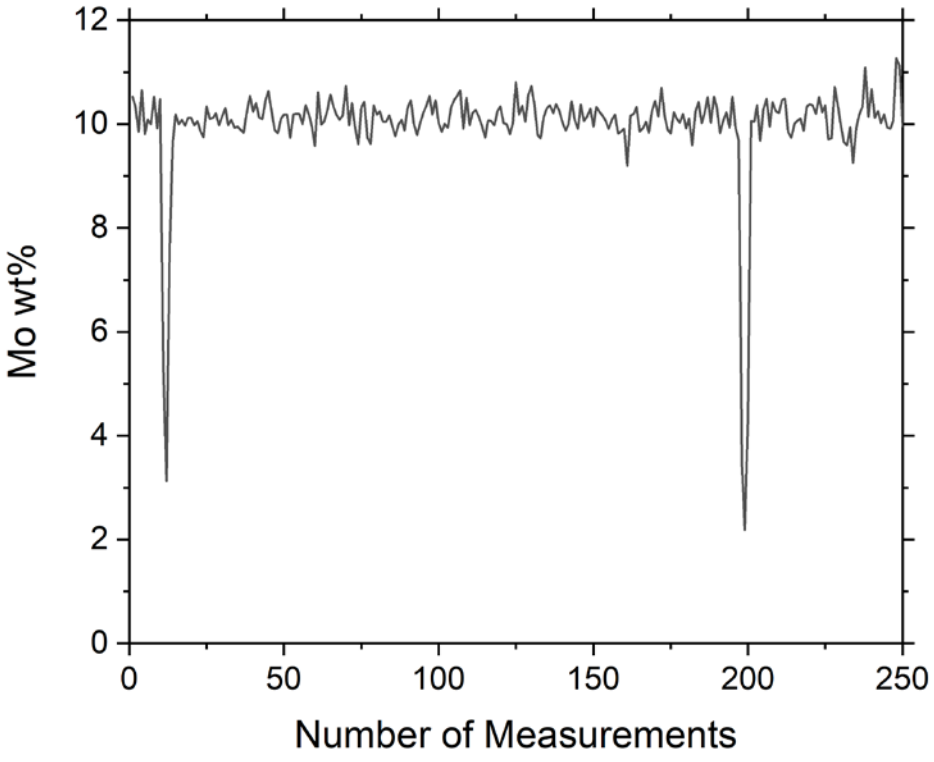

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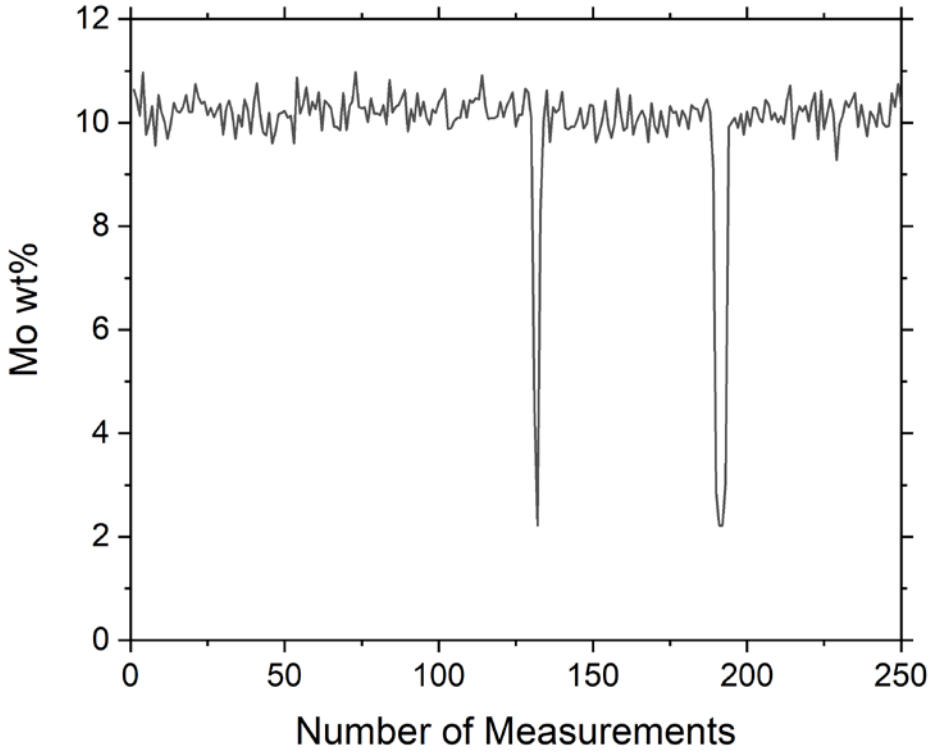
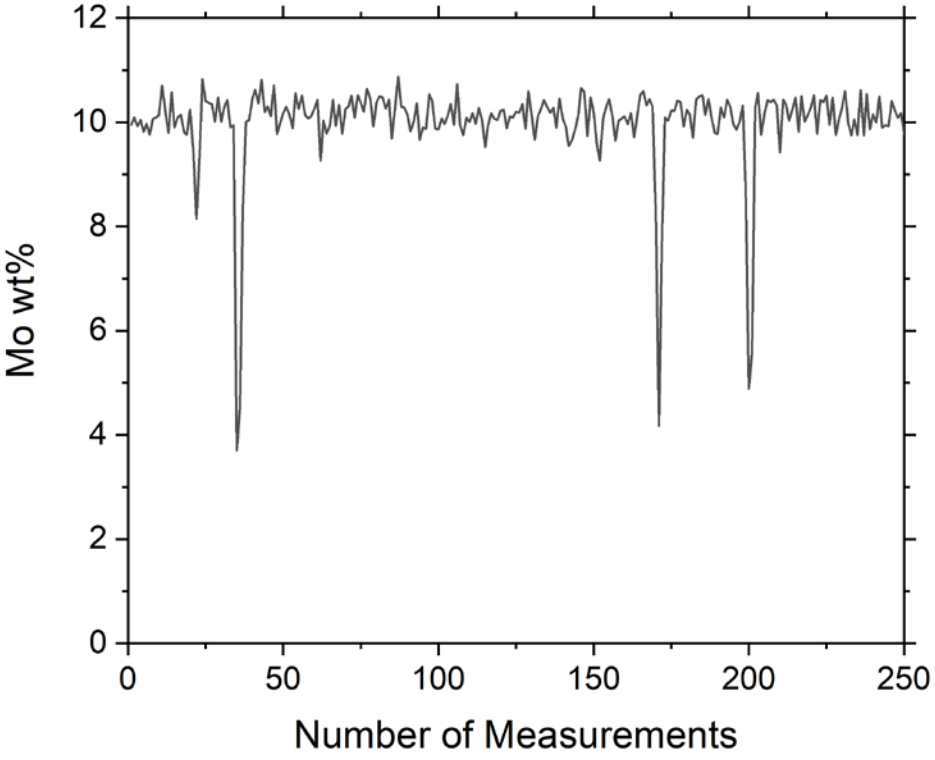


(c)



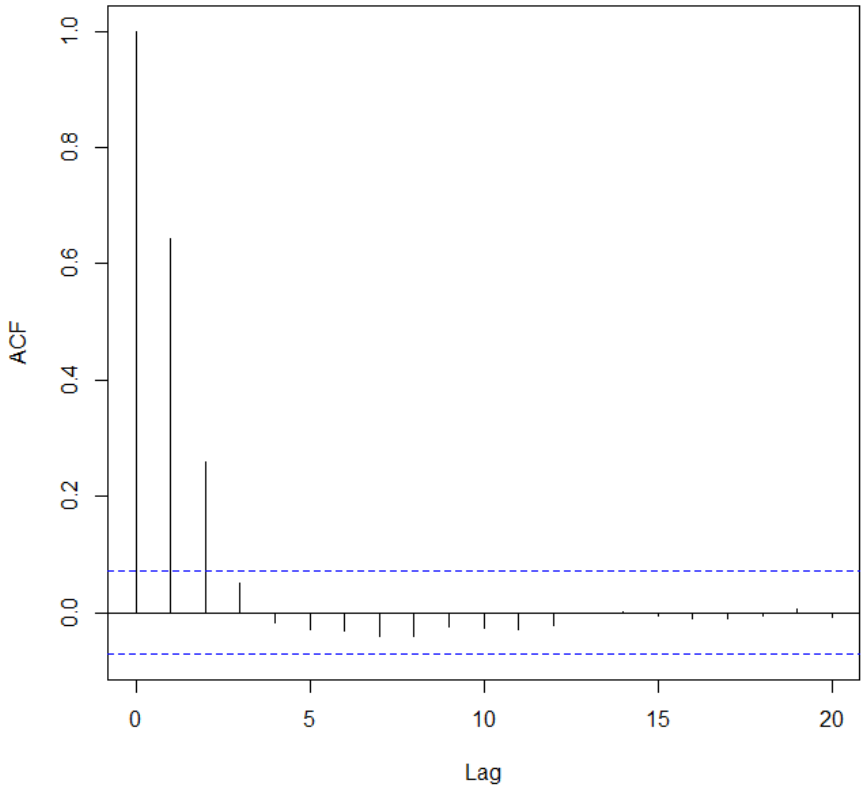
(d)

Figure 7. SE Image (a) with Top (b), Middle (c), and Bottom (d) EDS Line Scans for Plate 2

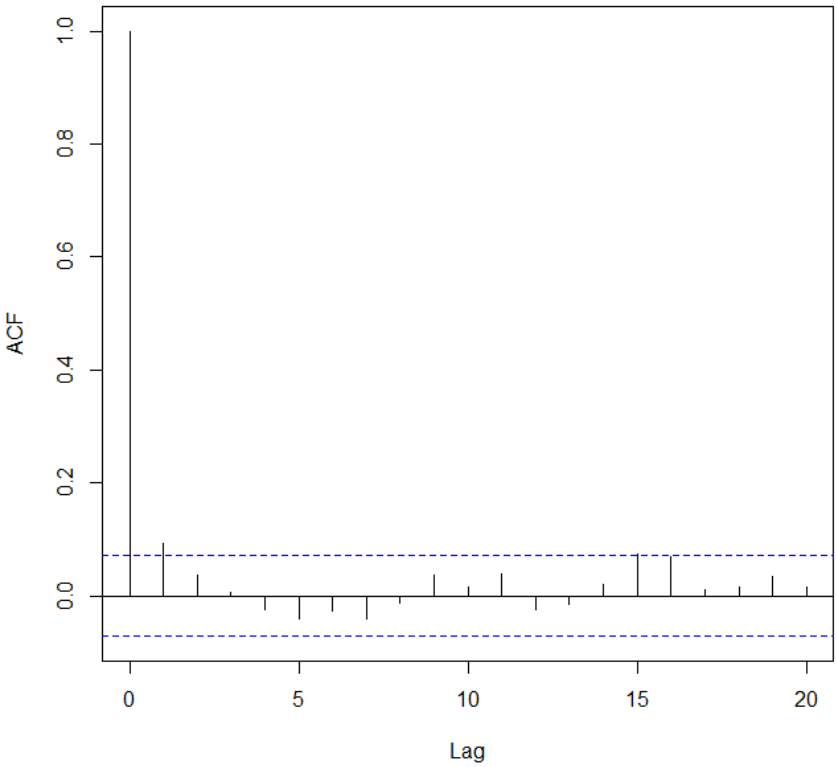
Table 4. Effect of Sample Size on Plate Qualification for Plate 2

N	**5**	**10**	**20**	**50**	**100**	**200**
Repetition	1000	1000	1000	1000	1000	1000
k_2	5.09	3.38	2.75	2.37	2.23	2.14
Mean (s)	0.26	0.28	0.28	0.29	0.29	0.29
Mean(k_2s)	1.36	0.96	0.78	0.69	0.64	0.62
Lower Bound (L)	8.64	9.34	9.22	9.31	9.36	9.38
Upper Bound (U)	11.36	10.96	10.78	10.69	10.64	10.62

A spatial independence study using ACF was also conducted for Plate 2. The critical lag distance for scenarios both with and without particles are similar and close to 5 µm (Figure 8). The ACF results are in line with the observation from Figure 7, which shows no clear pattern of data distribution except white noise.



(a)



(b)

Figure 8. ACF Results for Plate 2 with (a) and without Particles (b)

4.0 Recommendation on the Minimum Number of Mo wt% Measurements to Determine Fuel Plate Qualification

It was shown in the second example that 10 measurements are already sufficient for Plate 2 to be qualified. However, this number can be different for other areas within a plate (or for a different plate) with different degrees of homogenization. Next, we provide a general recommendation for the optimal number of measurements to determine fuel plate qualification if no available measurement data exist in advance. As mentioned earlier, Eq. (10) indicates that $k_2(N,\gamma,P)$ is only a function of sample size N with fixed $P = \gamma = 0.95$. A plot of k_2 versus N in Figure 9 shows that k_2 gradually decreases (black line) but its slope slowly increases (red line) with increasing N. The value of k_2 eventually approaches an asymptote of ~2. A gradient of k_2 that is greater than −0.1% can be considered a mathematical indication of stability. The corresponding value of N at $\frac{dk_2}{dN} = -0.1\%$ is 133, and the associated measurement time (~2 h) is experimentally affordable. Therefore, it is reasonable to select $N = 133$ as the suggested number of measurements to determine the fuel plate qualification, resulting in k_2 = 2.19.

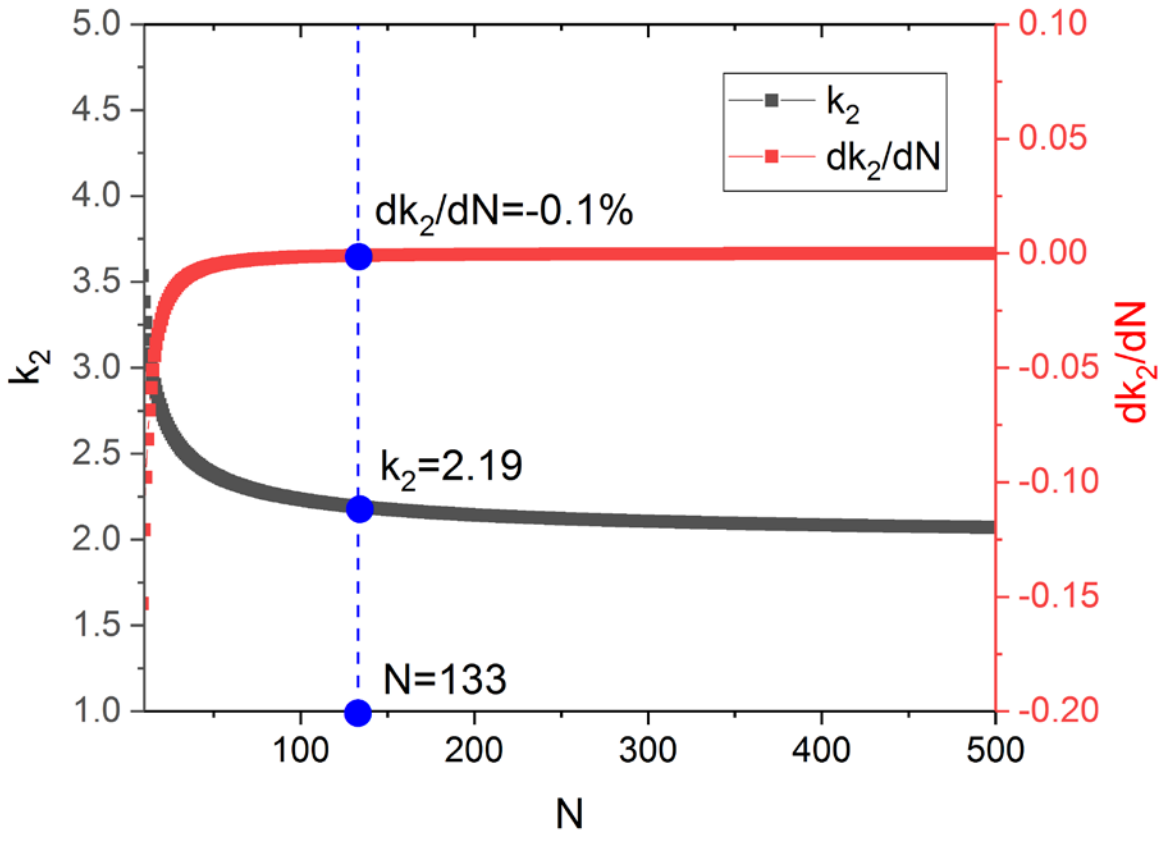


Figure 9. Relationship of k_2 and its Gradient with N

A reasonable conclusion of from Eq. (8) in this application is that the 95/95 TI problem simplifies to the criterion $k_2 s \leq 1$ for a plate to satisfy qualification requirements, if our assumption of average 10 wt% Mo is true. The proposed number of independent Mo weight percentage measurements should be at least 133 across the longitudinal cross section, and that determines $k_2 = 2.19$. Discard the measurements if Mo weight percentage is outside the range of 7–13% to avoid influence from carbides. To make sure the selected data are not autocorrelated and are independent, a suggested procedure of data collection is described as follows. (1) Collect the initial 133 EDS line-scan data with a normal 1 μm spacing between measurements. (2) Conduct the ACF analysis, and then determine the critical lag distance. (3) Determine the number of valid collections of data points among the original 133 data and then make additional collections based on the critical lag distance obtained from Step 2. After collecting sufficient amount of data points, the given fuel plate can be considered a qualified piece and be supplied to the subsequent processes only if $s \leq 0.46$, which is equivalent to $k_2 s \leq 1$. Otherwise, the plate is disqualified.

5.0 Conclusion

To characterize the quality of U-10Mo in casting is important and also challenging. A statistical approach based on the concept of TI was implemented to determine fuel plate qualification. This study also recommends a minimum number of required Mo wt% measurements to represent the distribution behavior of Mo for the entire fuel plate when other data are not available to determine the number of samples needed. Then the given qualification specification can be simplified using only sample variance under reasonable assumptions. In particular, for the fuel plates discussed here, at least 133 data points across the line scans are sufficient. Data points for which the line scan intersects a carbide, or a secondary-phase particle are to be discarded. The collection of data must be independent without any correlation, to assure that the collected measurements are a valid representation of the entire fuel plate. For sample standard deviation values equal to or less than 0.46 and means equal to 10 wt% Mo, the given plate will ideally be qualified, whereas for standard deviation values higher than 0.46, the material is not qualified. By monitoring fuel plate quality using the proposed method, the efficiency of the plate qualification is greatly enhanced and fabrication costs are reduced. The developed methodology can be extended to other fields and serve as a metric for quality control.

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