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X-ray Gauge Monitoring of LLNL Replica Carbon Production Process

by

Daniel L. Lewis

This report contains results of a series of x-ray gauging measurements performed on bars tracked through the LLNL replica carbon production process.

We were tasked with monitoring a few salt bars through the entire production process to identify any possible problem areas. The process consists of five major steps:

- 1) Pressing fine sodium chloride crystals into a uniform salt bar,
- 2) Sintering the salt bar,
- 3) Infusing the bar with a phenolic polymer solution and convection oven drying it to remove the acetone solvent,
- 4) Carbonizing the polymer, and
- 5) Extracting the salt with water and freeze-drying the final carbon foam bar.

After pressing the salt bars, six typical bars were selected for characterization: LS1875, LS1876, LS1879, LS1881, LS1883, and LS1886. These six bars were to be gauged after each of the five production steps to check for any inhomogeneity introductions. Two of the bars, however, were damaged during the final production step, resulting in complete process information for only four of the six bars. This report contains only the information gathered for these four bars. The data obtained for the other two bars, prior to damage, showed no significant difference from the data presented here.

The x-ray gauge was used to determine the homogeneity of a bar by measuring the photon transmission through its thickness and converting it to areal density. The areal density was calculated from the transmission measurement, the relative abundance of the elements present, and the elements' tabulated cross section values¹. The density and composition of the bars at various stages of processing required that we use two different photon sources. Differences in scattering and errors in cross section values may introduce small errors in the calculated areal densities. Although these errors may affect the absolute areal density value by several percent, they are negligible when normalized to the bar's mean areal density. Therefore, all gauging results are presented as a percent deviation from the mean areal density of the bar.

The studied bars were approximately 4.5" long by 1.0" wide by 0.5" thick and were to be gauged through the thickness dimension. Each bar was gauged at 32 points in a uniform matrix as shown in Figure 1. We gauged the bars in the same location after each process step so that subsequent results could be compared. The gauge counting time was chosen to obtain one-half percent counting statistics precision in the results. The salt, with a bulk density of about 1.4 g/cm³, required the use of an americium-241 radioisotope source that produces 59.5 keV photons. The americium-241 source was used for gauging

¹E. F. Plechaty, D. E. Cullen, and R. J. Howerton, *Tables and Graphs of Photon-Interaction Cross Sections From 0.1 keV to 100 MeV Derived From The LLL Evaluated-Nuclear-Data Library*, Lawrence Livermore Laboratory, Livermore, CA UCRL-50400, Vol. 6, Rev. 3 (1981).

after the first four process steps. The final replica carbon foam bar, with a bulk density of about 50 mg/cm³, required the use of an iron-55 source that produces 5.9 keV photons.

Figure 1 - Layout of Gauging Point Locations

The first step was to press the fine sodium chloride crystals into salt bars. The gauging results showed that the pressing of the salt bars was a very uniform process. The density variations were only about $\pm 2\%$ as can be seen from the contour plots of Figure 2. The plots were generated using a software package called WINGZ with each intersection on the plot representing a gauged point and the intermediate integration and shading being performed by the software. The plots do not show any definite trends in the data as to the locations of higher or lower areal density regions. Table 1 summarizes the areal density gauging results. The pressed salt bars are very uniform in areal density.

Table 1
Salt Bar Areal Densities After Pressing

Bar ID#	Mean Areal Density (g/cm ²)	Standard Deviation (%)	Min -> Max Range (%)
LS 1875	1.542	0.724	2.270
LS 1881	1.626	0.391	1.722
LS 1883	1.627	0.523	1.967
LS 1886	1.525	0.608	2.624

The second step consisted of sintering the salt bars to fuse the salt crystals together. The results can be found in the plots of Figure 3. They show no significant change in the homogeneity of the bars. Table 2 summarizes the areal density gauging results and shows that there was a slight density increase due to the shrinkage of the bars.

Table 2
Salt Bar Areal Densities After Sintering

Bar ID#	Mean Areal Density (g/cm ²)	Standard Deviation (%)	Min -> Max Range (%)
LS 1875	1.570	0.573	2.624
LS 1881	1.655	0.477	1.970
LS 1883	1.652	0.512	2.124
LS 1886	1.549	0.641	2.337

Next, the bars were infused with a phenolic polymer solution and then convection oven dried to remove the acetone solvent. We gauged the bars and reduced the data as if all the x-ray attenuation was due to salt. The contour plots of Figure 4A show that a definite density pattern has developed in the bars. They indicate that the areal density is lower in

the center and higher at the edges of the bars. Examination of Table 3A shows that the average areal density of the bars increased due to the addition of the polymer, as expected, and also, more importantly, that the density variation increased. Photon attenuation is a very strong function of Z , atomic number. It is approximately proportional to Z^4 . Thus, interpreting the additional attenuation arising from the polymer, (containing carbon, hydrogen, and oxygen), as if it were sodium chloride does not provide a useful estimate of the uniformity of the polymer.

Table 3A
Areal Densities After Polymer Infusion (Polymer & Salt)

Bar ID#	Mean Areal Density (g/cm^2)	Standard Deviation (%)	Min -> Max Range (%)
LS 1875	1.644	0.685	2.481
LS 1881	1.720	0.697	3.360
LS 1883	1.718	0.668	3.190
LS 1886	1.618	0.775	3.004

We separated the attenuation caused by the polymer from the attenuation caused by the salt. Since the gauge measurements were made in the same spots, we subtracted the data obtained from the previous gauging step. The assumption was made that this operation would remove all density variations due to the salt bar itself leaving only the contributions of the added polymer. This data was then converted to polymer areal densities and the results can be found in the plots of Figure 4B and in Table 3B. The areal density variations are extremely large and indicate that the infused polymer distribution is not very uniform. However, these results are susceptible to large errors due to the manner in which they were obtained and the fact that the polymer is practically transparent to the gauging photon energy. The uncertainty, based on standard counting statistics, was calculated to have a standard deviation of about 30 percent. Therefore, the conclusion from this data is that the polymer areal densities vary $\pm 45\%$ with 30% uncertainty, indicating the presence of real density variations in the polymer, but of an uncertain magnitude.

Table 3B
Areal Densities After Polymer Infusion (Polymer Only)

Bar ID#	Mean Areal Density (mg/cm^2)	Standard Deviation (%)	Min -> Max Range (%)
LS 1875	137.575	12.534	56.314
LS 1881	121.085	16.555	63.710
LS 1883	121.923	19.788	94.545
LS 1886	127.329	19.705	70.007

After the bars had been carbonized, we gauged them and, again, corrected for the attenuation of the salt. The residual attenuation was interpreted as arising only from the carbon. This data is shown in Figure 5 and Table 4. The areal density contours of the carbon within the salt are very similar to that observed for the polymer on the previous step. These results are subject to large statistical uncertainty as in the previous step and, in addition, there appears to be an added influence from further densification of the salt during carbonization.

Table 4
Areal Densities After Polymer Carbonization (Carbon Only)

Bar ID#	Mean Areal Density (mg/cm ²)	Standard Deviation (%)	Min -> Max Range (%)
LS 1875	193.825	15.217	53.327
LS 1881	198.260	13.371	58.825
LS 1883	210.066	17.083	77.839
LS 1886	198.782	14.760	65.143

The fifth and final step in the process uses water to extract the salt from the bar. It is then freeze-dried to obtain the finished replica carbon foam bar. The gauge results can be found in the plots of Figure 6 and in Table 5. These plots indicate, undoubtedly, that a substantial density gradient from center to edge exists in all finished carbon bars of this study.

Table 5
Areal Densities of the Final Replica Carbon Foam

Bar ID#	Mean Areal Density (mg/cm ²)	Standard Deviation (%)	Min -> Max Range (%)
LS 1875	55.581	8.888	31.485
LS 1881	55.109	9.932	33.388
LS 1883	55.493	9.961	30.634
LS 1886	58.493	8.221	28.892

In conclusion, the x-ray gauge results indicate that the polymer infusion process for these bars is extremely non-uniform. Large density gradients of the polymer in the bars ultimately results in large density gradients in the final product. The gauge reports that the center to edge gradients are on the order of 30% with less than 1% uncertainty. In addition, it must be remembered that these results are strictly two-dimensional. The variation could be much worse in three dimensions depending on the geometry of the density variations. A computed tomography study of the density variation in three dimensions is currently underway for these carbon bars.

Figure 2 - Uniformity of Salt Bars After Pressing

Figure 3 - Uniformity of Salt Bars After Sintering

Figure 4A - Uniformity After Polymer Infusion (Polymer & Salt)

Figure 4B - Uniformity After Polymer Infusion (Polymer Only)

Figure 5 - Uniformity After Polymer Carbonization (Carbon Only)

Figure 6 - Uniformity of Final Replica Carbon Foam

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