

# LCLS Heavy Met Outgassing Tests\*

K. I. Kishiyama, D. M. Behne

Lawrence Livermore National Laboratory

## Abstract

A Heavy Met that is 95% tungsten, 3% nickel and 2% iron and sintered to 100% density and is Ultra High Vacuum (UHV) compatible is proposed for use as the X-ray slit in the Front End Enclosure and the Fixed Mask for the Linac Coherent Light Source (LCLS). The Heavy Met was tested in the LLNL Vacuum Sciences and Engineering Lab (VSEL) to determine its outgassing rate and its overall compatibility with the vacuum requirements for LCLS.

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## **Test Report**

### **LCLS Heavy Met Outgassing Tests**

Keith Kishiyama, LLNL  
Dan Behne, LLNL

#### **Introduction**

A Heavy Met that is 95% tungsten, 3% nickel and 2% iron and sintered to 100% density and is Ultra High Vacuum (UHV) compatible is proposed for use as the X-ray slit in the Front End Enclosure and the Fixed Mask for the Linac Coherent Light Source (LCLS). The Heavy Met was tested in the LLNL Vacuum Sciences and Engineering Lab (VSEL) Outgassing Measurement Test Stand #2 in building 132.

Determining the outgassing rate of the Heavy Met is essential to understanding the total gas load of the vacuum system, which in turn determines the pumping necessary to meet the required vacuum pressure. Figure 1 shows one of the Heavy Met samples.

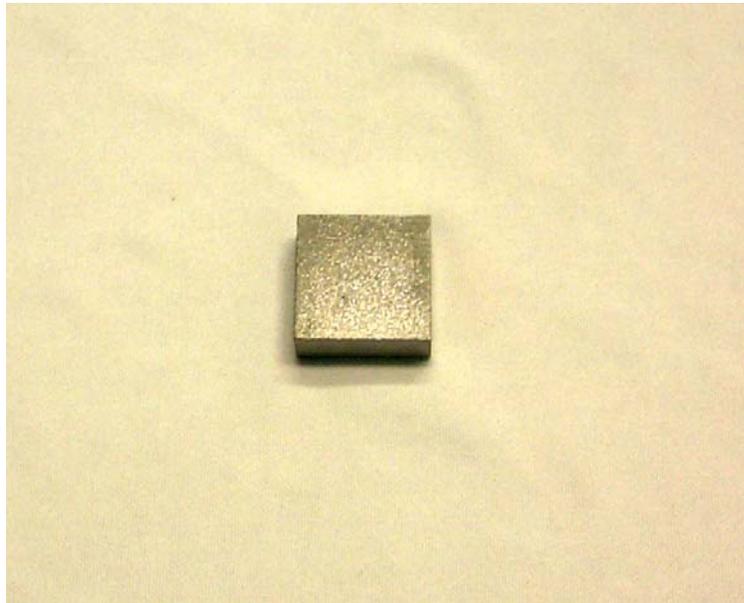


Figure 1. LCLS Heavy Met sample.

The VSEL Outgassing Measurement Test Stand #2 is shown in Figure 2. The test stand is a vacuum vessel divided into two chambers. The wall that divides the vessel into two chambers has a fixed

diameter orifice. The chamber that is designed to hold the sample has an 8 inch diameter conflat flange for inserting the sample. The instrumentation on the sample chamber consists of a MKS Baratron, a Granville Phillips Model 370 Stabil-ion gauge and a Granville Phillips Convector gauge.

The actively pumped secondary chamber on the Outgassing Measurement Test Stand is pumped by a Seiko Seiki magnetically levitated turbomolecular pump. All of the valves are full metal sealed valves. The instrumentation on the pumped chamber consists of a Granville Phillips Model 370 Stabil-ion gauge, a Granville Phillips Convector and a Leybold-Inificon Transpector RGA.

Each Granville Phillips Model 370 Stabil-ion gauge is individually calibrated at the factory and is shipped with a memory module containing the calibration. The memory module is then downloaded into the gauge controller and provides 3% accuracy.

The convector, stabil-ion gauge and RGA data is recorded by a PC running Labview software. The software calculates the outgassing rate from the differential pressure across the orifice using the pressure readings from the two stabil-ion gauges.

The outgassing rate of the chamber itself has been measured many times and the system has been tested with a calibrated leak ( $Q = 2.1 \times 10^{-5}$  Torr-Liters/sec). The background outgassing rate of the chamber is subtracted from the total measured outgassing rate to yield  $Q$ , the total outgassing rate of the sample or in this case the calibrated leak. The result with calibrated leak was within 10%. This error is within reason since each Stabil-ion gauge has a 3% error. On top of the accuracy of the gauging, there are numerous other factors that can be sources of error in this type of measurement [1]. For the calibrated leak, the measured value was three orders of magnitude greater than the background, which helps to reduce some of the sources of error.

Dividing  $Q$ , the total outgassing rate by the surface area of the sample provides  $q_d$ , the specific outgassing rate. As discussed above, this technique works adequately with outgassing rates a few orders of magnitude above the background. There are other techniques that can be used to measure ultra low outgassing rates. [2]

The sample chamber is a cube where each side is 6 inches. Along with the additional surface area for the top sample insertion port and the instrumentation ports, the surface area is  $1464 \text{ cm}^2$ . The outgassing rate of the sample chamber alone after a 200 degree C bakeout is generally about  $3 \times 10^{-8}$  Torr-Liters/sec. This corresponds to an outgassing rate of  $2 \times 10^{-11}$  Torr-Liters/sec/ $\text{cm}^2$ , which agrees with published values for vacuum baked stainless steels [3].

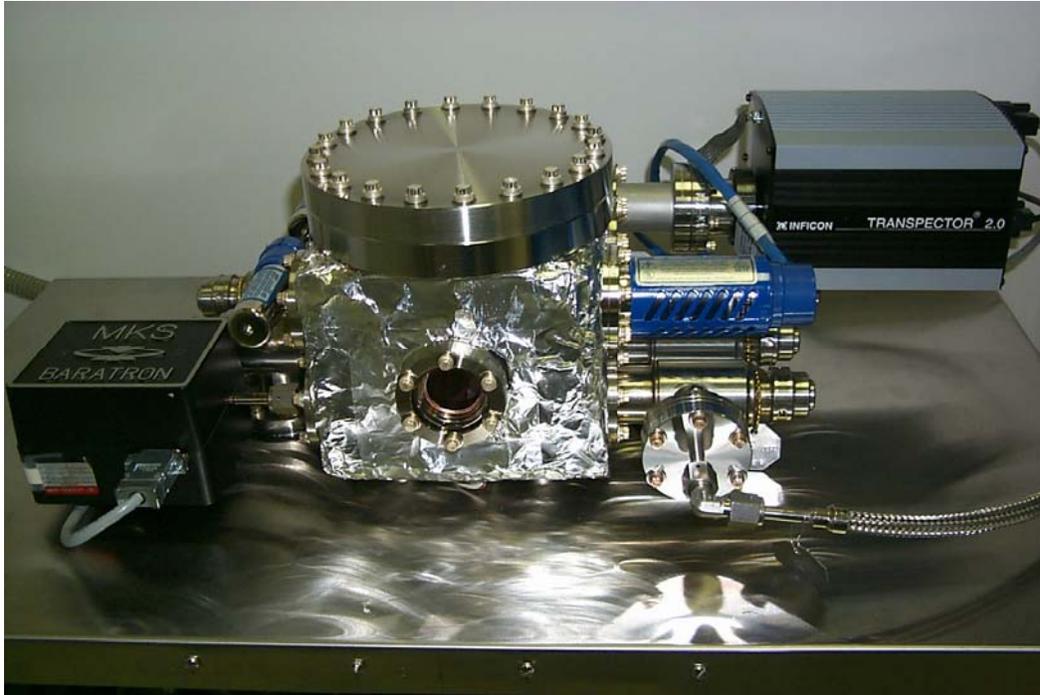


Figure 2. LLNL VSEL Outgassing Measurement Test Stand #2 in B132.

### Results

The 12 Heavy Met coupons were wiped down with a lint free cloth with 100% isopropyl alcohol and then placed in the outgassing measurement test stand and pumped under vacuum for 90 hours. The total surface area of the 12 coupons was  $243 \text{ cm}^2$ . The total outgassing rate at the end of the test was  $4.5 \times 10^{-8}$  Torr-Liters/sec. Subtracting the background outgassing rate of the chamber gives a total outgassing rate  $Q$  for the 12 sample coupons of  $1.5 \times 10^{-8}$  Torr-Liters/sec. A plot of the sample pressure and total outgas rate is shown in Figure 3. At the time the test was terminated, it appeared that the outgassing rate had leveled off to a constant rate.

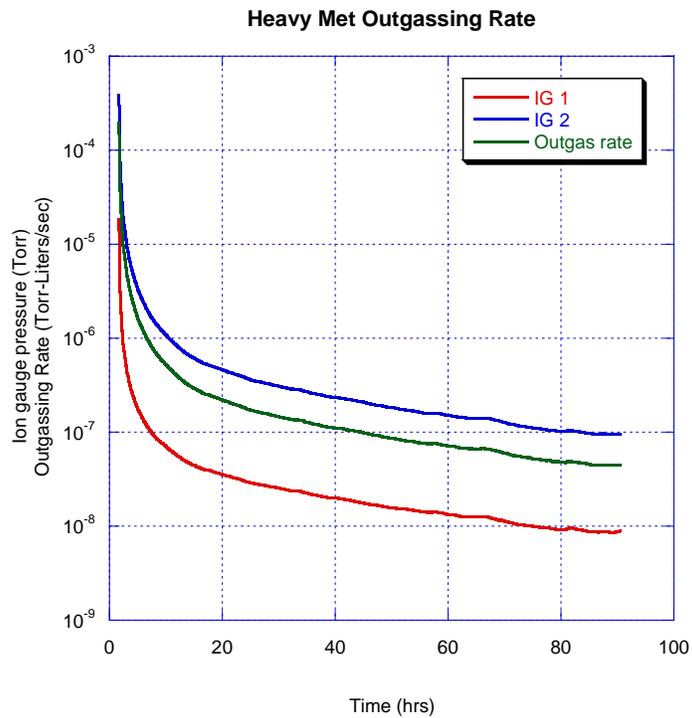


Figure 3. Outgas rate and pressure of Heavy Met

Figure 4 shows the spectrum from the Heavy Met after it had been under vacuum for a little over 90 hours and just before the Test Stand was vented and samples removed. The spectrum is a plot of ion intensity versus AMU. The RGA has not been calibrated to give the partial pressure of gas species in Torr.

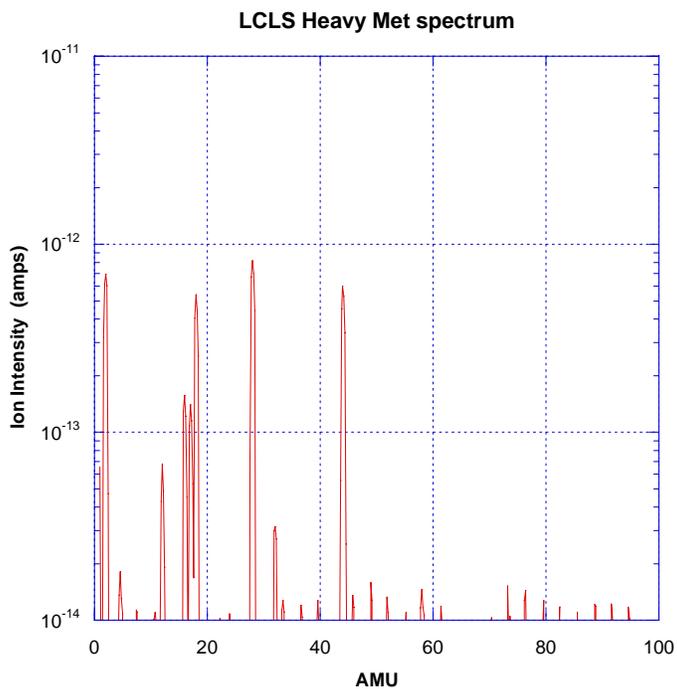


Figure 4. LCLS Heavy Met spectrum after 90 hours

Figure 5 shows a typical background spectrum of B132 Outgassing Measurement Test Stand after a 200 degree C bakeout. Once again, the spectrum is a plot of ion intensity versus AMU and the RGA has not been calibrated to give the partial pressure of gas species in Torr. However, both spectrums were taken using only the Faraday Cup detector and the electron multiplier turned off, so that the ion intensities on both spectrums are comparable.

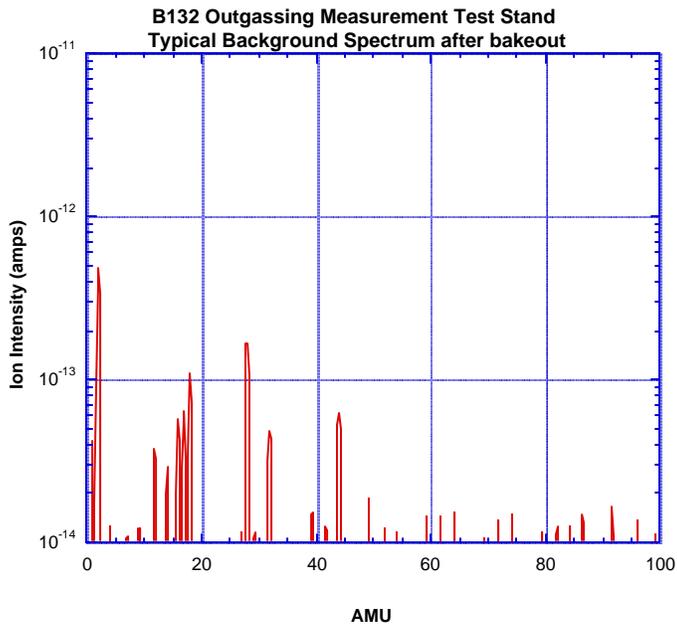


Figure 5. Typical background spectrum of test stand after bakeout

### Conclusion

When we subtract the background outgassing rate of the chamber, we get a total outgassing rate for the 12 sample coupons of  $1.50 \times 10^{-8}$  Torr-Liters/sec. Dividing by the total surface area of the samples gives a specific outgassing rate  $q_d$  of  $6 \times 10^{-11}$  Torr-Liters/sec/cm<sup>2</sup>.

Note that the background outgassing rate of the chamber represents 66% of the total measured outgassing rate. When subtracting the background rate from the total measured rate, we are subtracting two very small numbers and the difference is even smaller yet and thus a substantial error can result. Based on that, a conservative conclusion would be to say the specific outgassing rate  $q_d$  of the Heavy Met is  $\geq 1 \times 10^{-10}$  Torr-Liters/sec/cm<sup>2</sup>.

Based on the conservative value for the specific outgassing rate  $q_d$  of the Heavy Met, it can be concluded that the use of Heavy Met in the LCLS XTOD is compatible with the vacuum system.

It is recommended for future outgassing measurements for LCLS, that the test be designed such that the total outgassing rate of the sample is at least two orders of magnitude greater than the background outgassing rate. In the case of the Heavy Met, the surface area of the samples versus the surface area of the measurement chamber was only 17%. Increasing the surface area of the samples would give higher confidence in the results.

## References

1. Y. Strausser, *Review of Outgassing Results*, Varian Technical Note VR-51
2. K. Kishiyama, et al. *Measurement of Ultra Low Outgassing Rates for NLC UHV Vacuum Chambers*, Proceedings of the 2001 Particle Accelerator Conference, New York, (2001)
3. J. O'Hanlon, *A User's Guide to Vacuum Technology*, Wiley, New York, (1980)

## Footnote

The LLNL Vacuum Sciences and Engineering Lab has two other outgassing measurement test stands that are capable of testing larger samples. The VSEL also has a wide variety of vacuum furnaces. The largest furnace has a 16.5" conflat flange and has a maximum operating temperature of 700° C. This facility is available to support LCLS.