Next Generation Safe Delivery Source (SDS®4) Dopant Material Storage and Delivery Package

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INTRODUCTION

Sub-atmospheric gas storage and delivery systems, referred to as SAGS Type 1, were developed by Entegris and have been used to deliver gases in ion implant processes for many years under the trade name of SDS®. This technology stably and reversibly adsorbs pure dopant gases on an adsorbent substrate contained within a cylinder package. The SDS platform has been adopted industry wide and has proven to combine inherent gas cylinder safety with adsorption technology in an effective way to allow end users to safely deliver highly toxic materials, driving greater efficiency for ion implant processes. This poster includes key developments of SDS4, including:

- Factors and screening criteria of an adsorbent for electronic gas storage and delivery.
- Carbon adsorbent improvements for use in this application.
- Cylinder package hardware development and testing for unparalleled safety, purity, and reliability.
- Ion implant process validation.

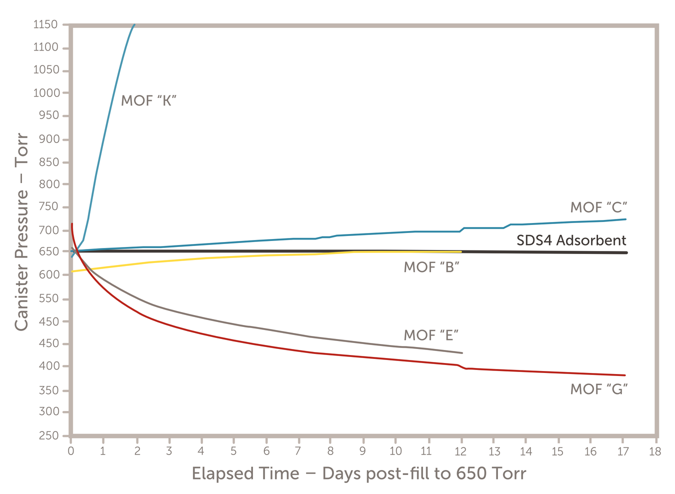
ADSORBENT SCREENING

In the development of SDS4, the landscape of microporous adsorbents was evaluated and assessed against various criteria, including:

- Density
- Toxicity
- Sensitivity to moisture
- Thermal instability
- Volume adsorption capacity
- Volume desorption retention
- Trace metal contamination
- Friability
- Particle generation
- Pressure instability
- Reactivity of adsorbent and adsorbate
- Cyclical capacity decay
- Manufacturability
- Cost

Entegris has selected and optimized the performance of monolithic microporous adsorbent carbon for SDS4.

Pressure Stability of PH_3 in 50 cc Adsorbent Canisters @21°C (post-fill to 650 Torr)



Visual Comparison of Various Adsorbents after Exposure to PH_3 and BF_3

Above left shows a comparison of the pressure stability of PH_3 on various adsorbents over time. No pressure change is observed with PH_3 adsorbed on SDS4 carbon over the entire testing time. Several other Metal Organic Framework (MOF) adsorbents display a significant pressure change, indicative of a reaction between the gas and adsorbent. In at least one MOF case the pressure goes super-atmospheric.

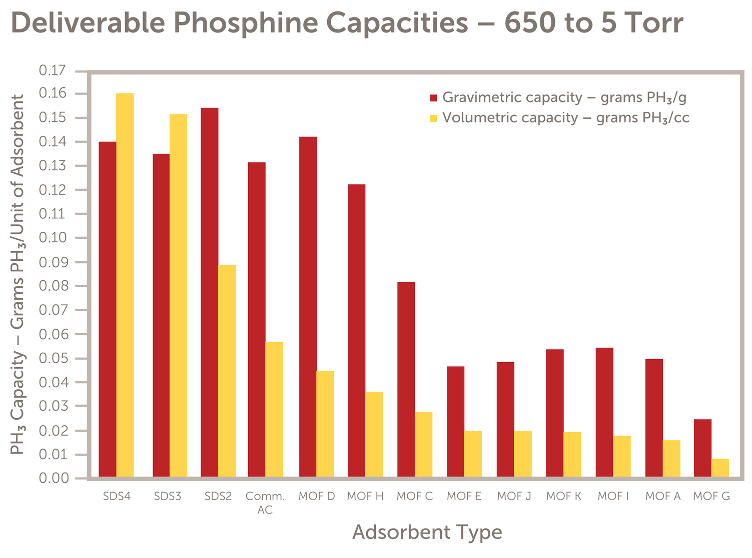
Above right displays the resulting color and form change of various MOF materials after this stability test. For each material the middle vial is the original state and then the resulting condition after exposure to either PH_3 (left) or BF_3 (right) is shown. Color and form changes are indicators of a reaction and resulting oxidation state change of the MOF transition metal. As can be seen the SDS3 material is stable.

KEY FEATURES

Capacity

When working within tight space constraints, such as the gas cabinet on an ion implanter, the optimal adsorbent for gas delivery is a high density microporous material. Having high surface area or gravimetric capacity is not enough if the density is low. Shown at right is the impact of bulk density on the volumetric PH_3 storage capacity.

Deliverable Phosphine Capacities – 650 to 5 Torr



Purity

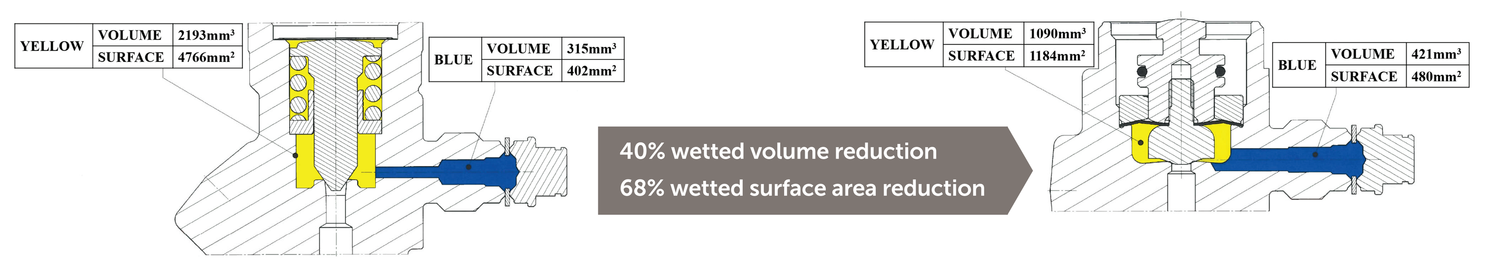
Purification methods and processes have been developed through an extensive series of tests and designed experiments. The continued use of robust and chemically inert carbon adsorbent common to previous SDS products, combined with advanced handling and processing techniques has enabled an extremely high final product purity of SDS4.

Trace metals analysis was performed by an independent 3rd party utilizing ICP-MS. In this test SDS4 adsorbent was tested alongside a zinc based MOF.

- No detectable amount of zinc in the SDS4 sample, however the MOF showed 13X the detection limit
- Test confirms the potential for the metal in the MOF framework to be volatilized and liberated from the gas cylinder which could cause contamination in the application

SDS4 cylinders are fitted with an ultra-high efficiency and low particle size retaining filter to mitigate particle shed. The broad capabilities of Entegris have been leveraged to develop this application-specific filter in-house.

The SDS4 valve features a tied-diaphragm valve, reducing the internal wetted components, volume, and surface area as compared to a standard diaphragm valve.



Safety

A key safety development for SDS4 is a visual valve open/closed indicator. Entegris has worked closely with the valve supplier to develop an application-specific valve state indicator for ion implant cylinders. Cylinders can be installed in either vertical or horizontal orientations in ion implant gas boxes. This indicator allows the user to view if the cylinder is opened or closed from the top or side orientation.

VALIDATION

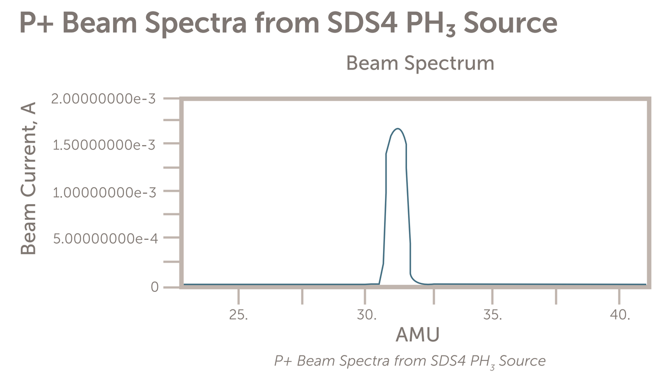
Various customer tests were performed to validate SDS4, including:

- No significant As+ and P+ beam current change from SDS3 to SDS4 (shown below left)
- No observed source glitching
- VPD-ICPMS analysis for trace elements matched control tool, shown right
- Sheet resistivity matched within 1 Ohm of control tool
- Particle monitoring has matched within 1.5% of control tool
- SIMS profiles matched within 2% of control tool (shown below right)

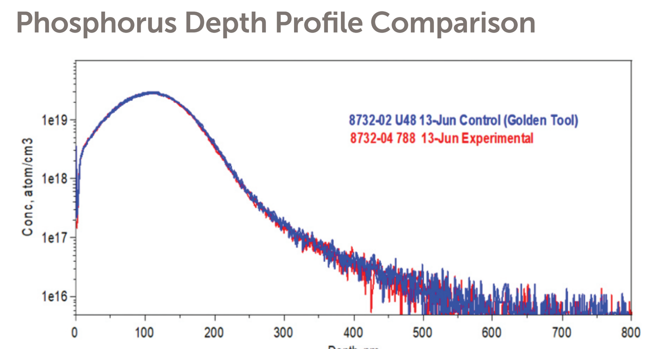
SDS4 Validation, VPD-ICPMS Measurement

Element	Atomic mass	Concentration (atoms/cm²)
Aluminum	27	2.40E+08
Chromium	52	8.10E+07
Copper	65	3.60E+08
Iron	56	1.30E+08
Magnesium	24	1.60E+08
Molybdenum	98	2.60E+07
Sodium	23	4.70E+08
Nickel	58	4.30E+07
Titanium	148	1.10E+09
Tungsten	184	2.10E+07
Zinc	66	3.40E+07

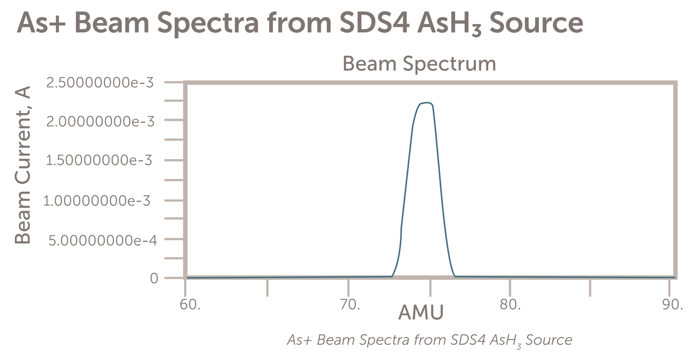
P+ Beam Spectra from SDS4 PH_3 Source



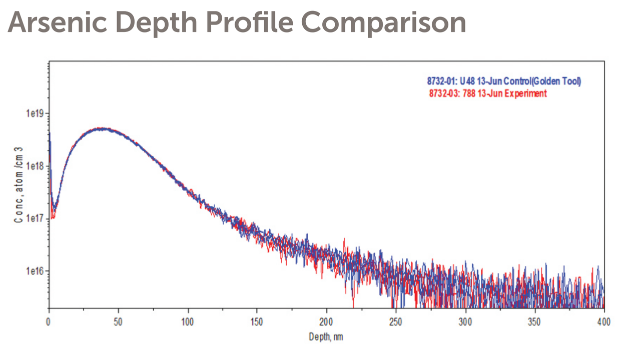
Phosphorus Depth Profile Comparison



As+ Beam Spectra from SDS4 AsH_3 Source



Arsenic Depth Profile Comparison



CONCLUSION

An array of microporous adsorbents were identified and screened for use in a SAGS I application. Results show that the SDS carbon-based adsorbent is the best candidate for this application. Advanced processing techniques enable a high final product purity. Package hardware is designed to further improve the safety, performance, and reliability of SDS4. Final product performance has been verified through external customer testing, confirming SDS4 exceeds ion implant application requirements.