Performance and Reliability of the Fourth Generation of Safe Delivery Source® (SDS®4) in the Ion Implantation Application

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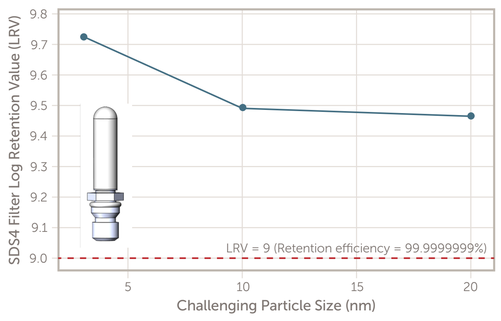
INTRODUCTION

Entegris has utilized its extensive subatmospheric storage and delivery (SAGS) application knowledge, combined with the broad capabilities of materials science and mechanical design to develop the fourth generation of the Safe Delivery Source® (SDS®4). With an improved particle filter, cylinder valve, and advanced carbon adsorbent purification process, the SDS4 cylinder package has been carefully designed to meet increasingly stringent demands on purity and safety of storing, handling, and delivering electronic dopant gases. The technical data detailed in this poster focuses on validating the performance and reliability improvement of the SDS4 hydride cylinder package. Several key performance indicators are characterized, including filtration efficiency, valve reliability, pressure and purity stability, flow rate capability, and gas deliverables over the entire cylinder life cycle. Overall product performance was validated in an ion source test stand to confirm that SDS4 has added unparalleled safety and performance to exceed ion implant application requirements.

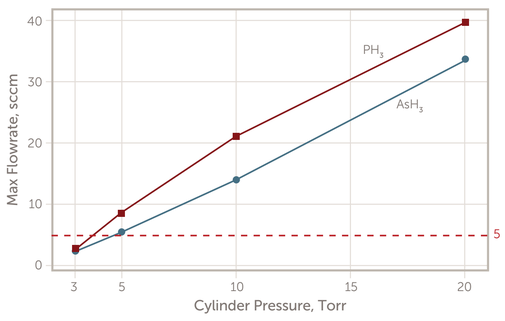
EXPERIMENT AND TEST RESULTS

SDS4 Filter Log Retention Value vs. Particle Size
Silver Nanoparticles; Flowrate = 0.3 SLPM (air)



Each data point is an average of at least three replicates

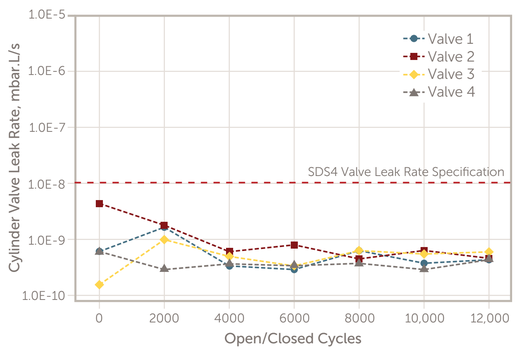
SDS4 Hydride Flowrate Capability vs. Pressure (<20 Torr)



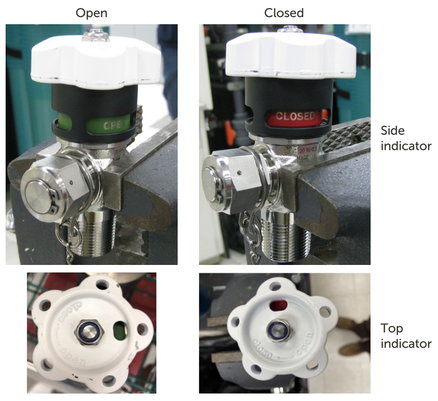
Each data point is an average of at least eight replicates;
Flow measurement: Brook GF120 XSD Mass Flow Controller

The gas microcontamination control expertise and technology of Entegris has been leveraged to design an application-specific ultra-high efficiency filter with particle size rating at 0.003 microns (3 nm). The filtration efficiency of the Entegris SDS4 3 nm filter was validated at a third-party testing center, following the test method and SEMI F38-0699. Testing has shown the Entegris SDS4 3 nm filter achieved target filtration efficiency (>9 LRV) across different particle sizes at the rated flowrate. The SDS4 filter greatly reduces particle contamination while still maintaining typical ion implant commanded flowrate at inlet pressures less than 5 Torr.

SDS4 Valve Leak Rate vs. Cycles
@ Entegris Valve Cycle Test System



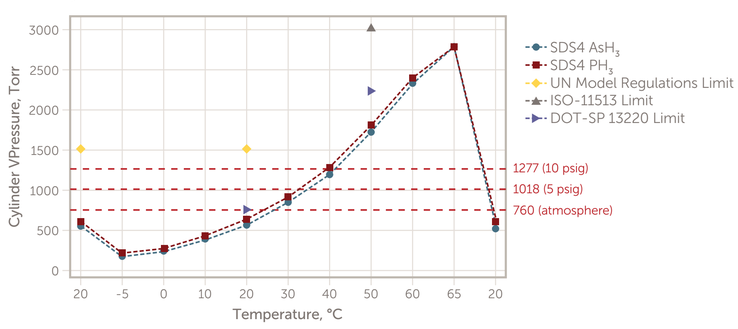
Closing torque = 7 N•m (62 in•lbs) and inlet presure = 609 psia of Helium



SDS4 valve open/closed indicator conditions after 12,000 cycles

The new application-specific SDS4 cylinder valve was designed jointly by Entegris and the valve manufacturer with unique specifications and features. The valve designs are tested by the manufacturer per existing CGA V-9 2012, ISO 10297:2014, and ISO 14246 standards. Entegris also established a complete incoming quality inspection procedure and sampling plan to evaluate the valve performance and reliability using an in-house valve cycle testing system (Entegris VCTS). The system was designed to repeatedly open and close cylinder valves at the rated operation conditions, effectively replicating the valve life span and providing insight into the long-term reliability. Valve leak tightness consistently outperformed the spec of <1.0E-8 mbar.L/s; averaging a leak rate of 3.9E-10 throughout 12,000 cycles.

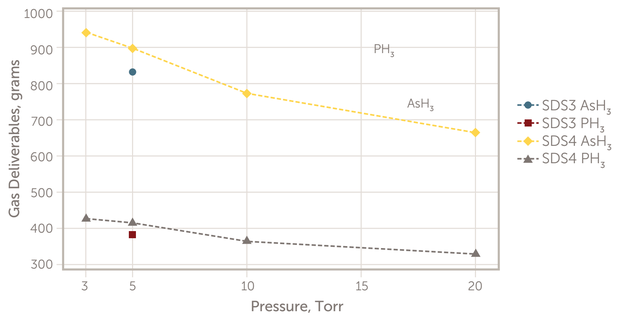
SDS4 Cylinder Internal Pressure vs. Temperature



Data collected from standard 2.5 liter (JY) size SDS4 cylinder

It is known that Type 1 SAGS cylinder pressure varies with temperature due to equilibrium effects between the gas phase in the cylinder head space and the solid phase in the carbon adsorbent. An experiment was conducted to characterize the relationship between cylinder pressure and temperature, ranging from -5 to 65°C. The results verified that the SDS4 product meets the definition of an Adsorbed Gas in U.N. model regulations and complies with ISO-11513 and DOT-SP 13220 Special Permit requirements for the sub-atmospheric gas delivery system. The temperature cycling results indicate that the gas physisorption equilibrium process within the package is entirely reversible and further confirming the chemical inertness of the SDS4 adsorbent.

SDS4 Hydride 2.5 L Cylinder Deliverables vs. Pressure (<20 Torr)
Lower bound of the 95% confidence interval plot

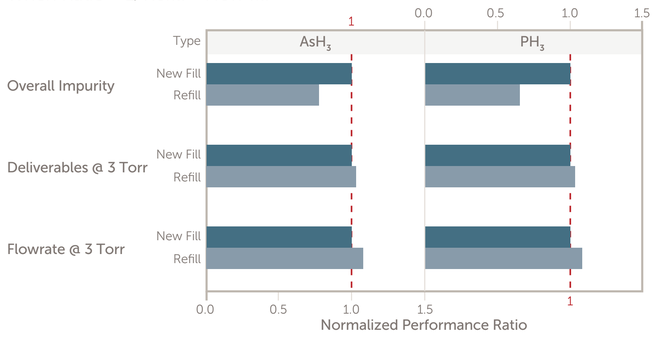


Each data point is an average of at least eight replicates

The SDS4 package not only increases gas purity but also allows the package to adsorb higher loadings of implant gases while the dimensions of the cylinder remain the same as the conventional product. The higher loadings and deliverables of the SDS4 package extend the service time and reduce the frequency of cylinder changes to yield better wafer production.

Parameters	Formula	Specification
Hydride Gas Purity	AsH_3/PH_3	≥99.985%
Carbon Dioxide	CO_2	≤25 ppm/v
Carbon Monoxide	CO	≤25 ppm/v
Methane	CH_4	≤25 ppm/v
Nitrogen	N_2	≤25 ppm/v
Oxygen + Argon	O_2 + Ar	≤25 ppm/v
Water	H_2O	≤25 ppm/v

SDS4 Hydride Cylinder Performance, New Fill vs. Refill
When Ratio > 1, Refill > New fill

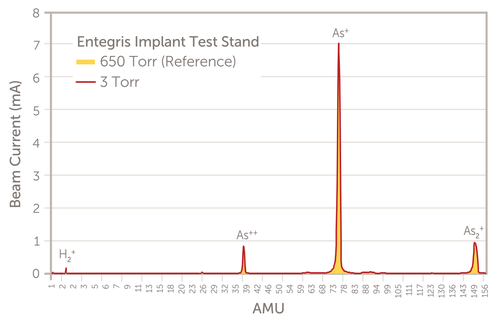


New fill performance is normalized to one for direct comparison;
Each data point is an average of at least three replicates
Panel variable: gas type

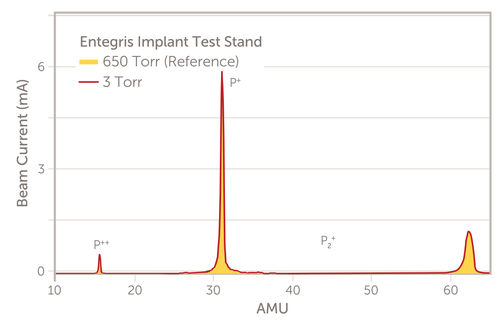
$$\text{Performance Ratio} = \frac{\text{Refill Performance}}{\text{New Fill Performance}}$$

Due to its rigid purity specification, the purified carbon adsorbent in the SDS4 package is very sensitive to any contaminant. It is essential to ensure the adsorbent has not been exposed to any unexpected contaminations before refilling the return SDS4 cylinders from end users. Upon return, the cylinders must undergo a complete return inspection, contamination detection, and refurbishment process to ensure the refill cylinders have equivalent performance characteristics to the new fills. The performance comparison between the new fill and refill SDS4 package show no statistically significant difference in purity between new fill and refill cylinders. The deliverables and flowrate at 3 Torr have equivalent performance to the new fill cylinders.

Mass Spectrum: SDS4 AsH_3 Dopant Package
Test conditions: Extraction = 20 kV, Gas flow = 1 sccm



Mass Spectrum: SDS4 PH_3 Dopant Package
Test conditions: Extraction = 20 kV, Gas flow = 1.5 sccm



The SDS4 Arsine and Phosphine performance was validated in the Entegris implant test stand to confirm the SDS4 cylinder packages have consistent beam performance and mass spectra for dopant fragments across full pressure ranges down to 3 Torr under typical ion implant recipe conditions, as shown in spectrums above; no beam glitching was observed.

CONCLUSION AND DISCUSSION

The performance and reliability of the Entegris SDS4 cylinder package has been validated by extensive internal and external product functional testing. Several key performance indicators are characterized to show that the SDS4 cylinder package has unparalleled safety, ultimate adsorbent stability, consistent gas purity, and gas deliverables across the entire package service life to meet the requirements of ion implant application and the semiconductor industry.