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Reliable Solid Precursor Evaporation for Advanced CVD and ALD Processes

Whitepaper
SEMPA SYSTEMS GmbH

Executive Summary

The semiconductor industry is currently witnessing a renewed focus on alternative metallization concepts. In particular, molybdenum based processes are gaining significant attention, driven by the need to reduce fluorine related side effects associated with conventional WF_6-based tungsten deposition. At the center of this transition is Molybdenum(VI)dichloride dioxide MoO_2Cl_2 – a solid precursor that combines excellent electrical properties with compatibility for advanced high-aspect-ratio structures.

However, the industrial adoption of MoO_2Cl_2 introduces a fundamental engineering challenge: the reliable and reproducible evaporation of solid precursors under high-temperature, high-flow, and safety-critical conditions.

SEMPA's VAPOR'BOT has been specifically designed to address these challenges. It provides a centralized, high-temperature solid precursor evaporation platform with integrated pump station, active temperature control, and precise delivery pressure regulation, enabling stable vapor generation for demanding CVD, ALD, and EPI processes.

This paper outlines the technological background, the specific challenges associated with MoO_2Cl_2, and the system architecture that enables industrial-scale deployment.

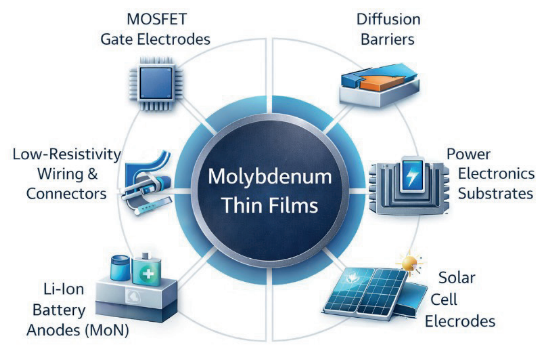
MoO_2Cl_2 – A Material in Strong Industrial Focus

The industry's shift toward alternative metal electrodes and barrier layers has intensified the search for fluorine-free process routes. Traditional tungsten deposition using WF_6 can leave fluorine residues, potentially affecting dielectric layers and interface stability in advanced nodes.

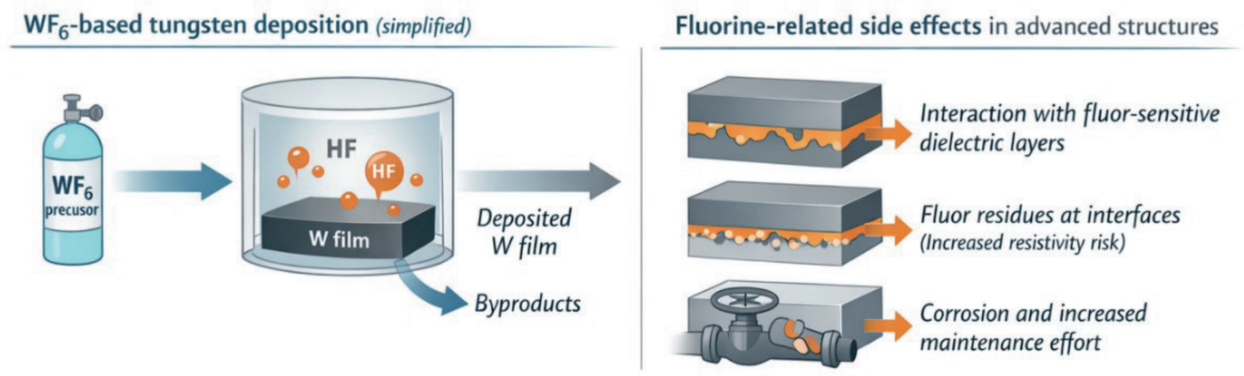
Molybdenum has emerged as a compelling alternative due to its:

- Low electrical resistivity
- Excellent thermal stability
- Strong compatibility with high-aspect-ratio structures
- Absence of fluorine in deposition chemistry

Beyond integrated circuits, Mo films are applied as diffusion barriers, photomasks, and substrates for power electronic devices. Furthermore, their role extends to renewable energy technologies, where they function as metal electrodes in solar cells to minimize energy loss, while molybdenum nitride variants are investigated as promising anode materials for lithium-ion batteries.



Among molybdenum precursors, MoO_2Cl_2 stands out as particularly promising.
It enables high-quality molybdenum film formation while avoiding the fluorine-related integration risks inherent in WF_6-based processes. As device scaling continues, this fluorine-free pathway is becoming increasingly relevant for advanced interconnects and electrode applications.



Yet, MoO_2Cl_2 is a solid precursor with low vapor pressure and elevated sublimation temperature requirements (Melting point = 176°C). Its successful industrial use therefore depends less on chemistry and more on evaporation technology.

The ability to generate a stable, precisely controlled vapor phase from MoO_2Cl_2 is now a decisive enabler for process integration.

The Engineering Challenge of Solid Precursor Evaporation

Unlike liquid precursors, solid materials introduce several interrelated challenges.

First, their vapor pressure is typically low, requiring elevated operating temperatures to achieve sufficient precursor flow. Second, temperature gradients within the canister or cabinet can cause inconsistent sublimation behavior. Third, desublimation in valves and transfer lines can lead to pressure instability, drift, or system blockage.
Finally, many chloride-based precursors are corrosive and potentially toxic, demanding robust material selection and comprehensive safety architecture.

In high-volume manufacturing environments, these issues directly impact:

- Process reproducibility
- Film uniformity
- Tool uptime
- Maintenance intervals
- Safety compliance

Consequently, solid precursor evaporation must be treated not as a peripheral subsystem, but as a core process platform.

The VAPOR'BOT Architecture – Designed for MoO_2Cl_2

SEMPA's VAPOR'BOT has been developed specifically for demanding solid precursor applications, including MoO_2Cl_2, WCl_6, $HfCl_4$, $GeCl_2$, $AlCl_3$, and $ZrCl_4$.
At its core, the system combines controlled high-temperature operation with precise pressure management and integrated safety features. Rather than relying on passive heating concepts, the VAPOR'BOT actively stabilizes the vapor generation process.

High-Temperature Operation for Low Vapor Pressure Materials

MoO_2Cl_2 requires elevated temperatures to generate sufficient vapor pressure. The VAPOR'BOT operates the cabinet in the 160–200 °C range where necessary, ensuring stable supply pressure conditions for demanding applications. High temperature capable components are selected to maintain reliability under these operating conditions.

Individual Heating Concept

One of the critical innovations lies in the separation of thermal zones. The canister is equipped with an individual heating jacket, while the cabinet itself functions as a controlled oven environment. This dual-level heating strategy reduces temperature gradients and prevents localized condensation effects.
By stabilizing the entire vapor path thermally, the risk of desublimation – a common issue with solid chlorides – is significantly reduced.

Integrated Valve Heating and Flow Optimization

Valves are integrated directly within the heated cabinet environment. This prevents cold spots that could otherwise promote precursor condensation. Large-dimension valves in process lines minimize pressure drop and enable higher flow stability.

Active Pressure and Temperature Control

Modern ALD and CVD processes operate within narrow pressure windows.
VAPOR'BOT incorporates active temperature adjustment based on canister fill level, ensuring stable supply pressure throughout the precursor lifecycle.

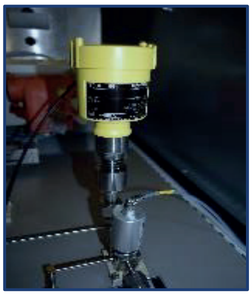
Delivery pressure can be controlled with a precision of ±50 Torr, which is particularly relevant for advanced process tools requiring tightly controlled partial pressures.

In addition, the system includes:

- High temperature pressure sensors
- Flow measurement including temperature monitoring
- Concentration monitoring for carrier gas applications

- Concentration monitoring for carrier gas application

- Flow measurement incl. temperature measurement



- High temperature pressure sensor

The result is a closed-loop vapor generation platform capable of maintaining stable MoO_2Cl_2 delivery over extended process runs.

Safety and Material Integrity for Chloride-Based Precursors

MoO_2Cl_2 and related chlorides require careful material compatibility and safety management.

VAPOR'BOT SOLID addresses corrosivity through the use of special alloy components instead of standard stainless steel. Toxicity risks are mitigated through cabinet ventilation, gas detection, and exhaust flow monitoring.

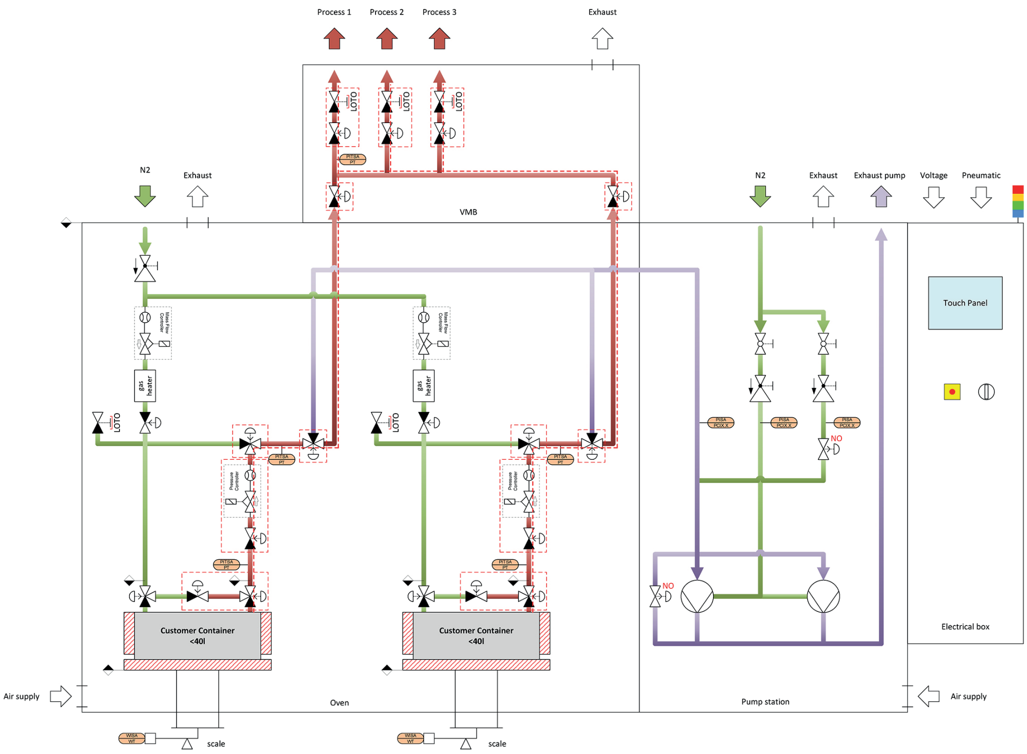
Overheat protection is ensured through multiple temperature sensors, software-based high-pressure relief functions, and accurate temperature control.
Operational reliability is further supported by emergency gas shutdown functionality, safety interlocks, multi-level user access control, PLC-based control with 12" touch screen interface, and SCADA communication capabilities.

These features make the system suitable for both R&D environments and high-volume manufacturing.

Industrial Integration and Efficiency

The VAPOR'BOT is designed as a complete system solution, including an integrated pump station. It supports canisters up to 40 L and can operate with argon or nitrogen as carrier or purge gas.

A particularly relevant aspect for cost efficiency is precursor utilization. The system enables more than 95% precursor utilization at canister change-over. For high-value materials such as MoO_2Cl_2, this significantly reduces operational cost and material waste.



Conclusion – Enabling the Transition to MoO_2Cl_2-Based Processes

MoO_2Cl_2 is increasingly recognized as a key precursor for fluorine-free molybdenum deposition in advanced semiconductor manufacturing. Its adoption is driven by integration benefits and electrical performance advantages. However, its solid-state nature and low vapor pressure make evaporation technology the decisive factor for industrial viability.

SEMPA's VAPOR'BOT SOLID provides a purpose-built solution for this transition. Through controlled high-temperature operation, active pressure regulation, thermally integrated flow paths, corrosion-resistant materials, and comprehensive safety architecture, it transforms solid precursor evaporation from a bottleneck into a stable process platform.

As molybdenum-based processes move further into industrial focus, robust evaporation systems will play a central role in enabling reliable and scalable production.

SEMPA SYSTEMS GmbH was founded in 2001 and has established itself as a reputable supplier of highest-purity gas and chemical supply systems, also for very special applications. Customers include major companies in the semiconductor and LED industries as well as representatives of the photovoltaic and pharma sectors. The company employs 200 people at its headquarters in Dresden and the subsidiaries.

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