

## Demonstration of AlScN growth using a high-temperature precursor supply unit for MOCVD equipment

SHIMIZU Yudai\* IKEJIRI Keitaro\*

### 1. Introduction

Nitride semiconductors deposited by metal organic chemical vapor deposition (MOCVD) are widely used in high-frequency and high-power devices, LEDs, lasers, and other applications due to their excellent physical properties. In recent years, it has become clear that the addition of rare-earth elements (such as Sc: scandium and Y: yttrium) during the deposition of nitride semiconductor films can produce superior properties. In particular, AlScN (aluminum scandium nitride), alloys of scandium with AlN has a ferroelectricity and is expected to contribute to lower power consumption of nonvolatile memory and higher carrier density in high electron mobility transistors (HEMTs)<sup>1)</sup>. This paper reports on the demonstration of AlScN deposition using Cp<sub>3</sub>Sc (tris(cyclopentadienyl) scandium), a low-vapor pressure precursor, using a high-temperature precursor supply unit developed by our company.

### 2. Technological challenges in supplying low-vapor pressure precursors

Magnetron sputtering, MBE, HVPE, and MOCVD have been reported as deposition methods for nitride semiconductors doped with rare-earth elements<sup>1)</sup>. The MOCVD is particularly useful in terms of crystal quality, deposition speed, thickness controllability, and mass producibility. However, there is a major problem in supplying rare-earth elements by the MOCVD method: the organic rare earth metal precursor has a low vapor pressure, making it difficult to vaporize and supply a sufficient amount needed to form a film. For example, the vapor pressure of Cp<sub>3</sub>Sc, one of the Sc precursors, is one to three orders of magnitude lower than that of conventional MO materials during their supply (near room temperature) even when the supply temperature is set above 100°C (Figure 1).

The heating mechanism of conventional precursor supply systems can only heat up to several tens of degrees Celsius and cannot vaporize the precursor sufficiently, making it difficult to supply low-vapor pressure precursors.

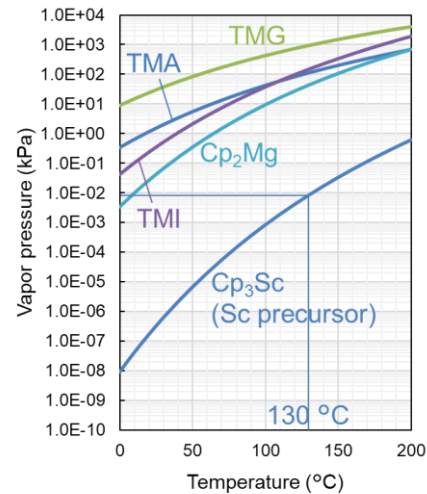


Figure 1 Vapor pressures of Cp<sub>3</sub>Sc and general MO materials

### 3. Features of SR4000-HT-LV

One of the pieces of our MOCVD equipment, the SR4000-HT, is widely used for deposition of GaN and AlN films up to 4 inches in size, and its greatest feature is that it is equipped with a triple-layer gas injector<sup>2)</sup>. This structure allows MO material and NH<sub>3</sub> to be introduced separately, suppressing parasitic gas-phase reactions of particulate generation. In addition, optimization of the gas flow by horizontal flow allows uniform film formation.

Based on the situation described in the previous chapter, we have developed a high-temperature precursor supply unit<sup>3)</sup> mainly for supplying rare-earth metal precursors to MOCVD equipment. This unit is capable of maintaining the entire precursor supply system at a high temperature of 130°C, and can stably supply low-vapor pressure precursors, which were difficult to supply by the conventional precursor supply systems. By incorporating this unit into the SR4000-HT, we realized MOCVD equipment SR4000-HT-LV, which enables crystal growth using low-vapor pressure precursors in MOCVD equipment.

\* Technology / R&D Dept., Compound Semiconductor Equipment Div., Innovation Unit

#### 4. Demonstration results

##### 4.1 Demonstration of AlScN deposition

We attempted AlScN deposition on GaN films using TMA as the Al precursor, Cp<sub>3</sub>Sc as the Sc precursor, and NH<sub>3</sub> as the N precursor, as shown in Figure 2. X-ray 2θ-ω scans revealed AlScN-derived peaks near the symmetry plane peaks of GaN (Figure 3). In addition, reciprocal lattice mapping of the (-1-124) plane showed an AlScN reflection above the GaN reflection, confirming that ideally lattice-matched AlScN was deposited on GaN (Figure 4). Also, the reflectance change during deposition indicated that the deposition rate of AlScN was about 70 nm/h.

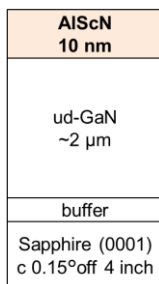


Figure 2 Film structure in this demonstration

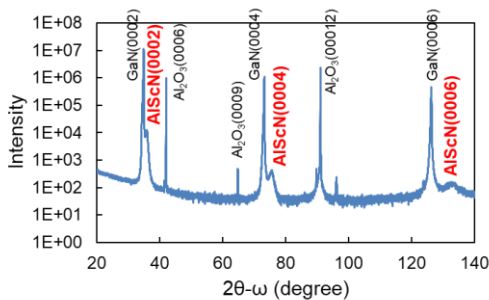


Figure 3 Result of X-ray 2θ-ω scans of AlScN film

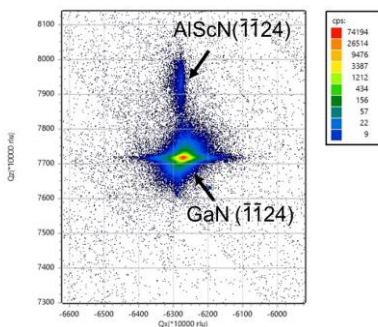


Figure 4 Reciprocal lattice mapping image of AlScN film

##### 4.2 Verification of memory effect

When different film types are deposited in the same deposition reactor, unintentional contamination of elements may occur in subsequent depositions due to residual precursors in the reactor from the previous growth. This is called the memory effect. By performing the deposit removal operation (H<sub>2</sub>+NH<sub>3</sub> baking and polishing), which is normally performed after GaN-based growth, after AlScN deposition, and then checking the Sc concentration in the ud-GaN layer using measurement by SIMS, the occurrence of the memory effect in this equipment was verified. As indicated in Figure 5, no Sc was detected in the subsequent GaN film, confirming that the memory effect caused by residual Sc in the system can be suppressed.

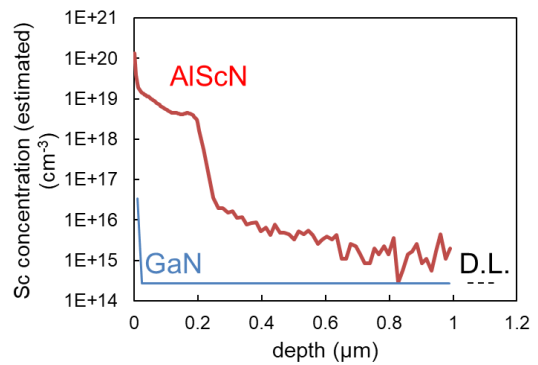


Figure 5 Depth profiles of Sc concentration in AlScN film (red) and subsequently deposited GaN film (blue), measured by SIMS

## 5. Conclusion

We succeeded in depositing AlScN films in which a low-vapor pressure precursor is used using the SR4000-HT-LV, and confirmed the formation of AlScN films lattice-matched to GaN. The deposition rate was about 70 nm/h, and it was confirmed that the influence of residual low-vapor pressure precursor on subsequent deposition can be suppressed. These results indicate that the SR4000-HT-LV can efficiently supply low-vapor pressure precursors by maintaining the entire supply system at a high temperature. The high-temperature precursor supply unit presented in this paper can be also combined with another piece of MOCVD equipment, such as the smaller SR4000-HT-RR, to achieve the same effect.

## Reference

- 1) S. Leone *et al.*, Phys. Status Solidi RRL 14, 1900535 (2020).
- 2) Kazuki Naito, MOCVD equipment SR4000HT for deep ultraviolet light emitting devices, Taiyo Nippon Sanso Technical Report No. 34 (2015)
- 3) Keitaro Ikejiri, Low-vapor pressure precursor supply unit (LV series) for MOCVD equipment, Taiyo Nippon Sanso Technical Report No. 42 (2023)