In situ monitoring of plasma etch processes with a quantum cascade laser arrangement in semiconductor industrial environment

N Lang¹,³, J Röpcke¹, H Zimmermann¹, A Steinbach² and S Wege²
¹INP Greifswald, Felix-Hausdorff-Str. 2, 17489 Greifswald, Germany
²Qimonda Dresden GmbH & Co. OHG, Königsbrücker-Str. 180, 01099 Dresden, Germany

E-Mail: lang@inp-greifswald.de

Abstract Concentrations of the etch product SiF₄ were measured online and in situ in technological etch plasmas with an especially designed quantum cascade laser arrangement for application in semiconductor industrial environment, the Q-MACS Etch. The combination of quantum cascade lasers and infra red absorption spectroscopy (QCLAS) opens up new attractive possibilities for plasma process monitoring and control. With the realization of a specific interface the Q-MACS Etch system is synchronized to the etch process and allows therefore automated measurements, which is important in a high volume production environment.

1. Introduction
Plasma technological procedures, like etching or deposition techniques play a key role for the industrial production of semiconductor devices. The challenge doing this can be found in the need of higher reproducibility when realizing structures on nanometer scales. In particular the multitude of discrete processing steps starting from the blank silicon wafer up to the final chip requires more and more methods of process monitoring to realize stable process control. Furthermore, the usage of molecular plasmas makes special demands for methods of in-situ process control.

Up to now, typically optical emission spectroscopy (OES), self excited electron resonance spectroscopy (SEERS) and mass spectrometry is applied for the online monitoring of plasma etch processes [1]. The drawback of all these methods is the lack of direct and quantitative access to etch relevant properties, like the concentration of precursors or of molecular etch products in the plasma bulk. A powerful diagnostic approach is given by infra red absorption spectroscopy with continuous sources (FTIR) [2] as well as with narrowband led salt lasers (TDLAS) [3,4] and quantum cascade lasers (QCLAS) [5-10]. The availability of quantum cascade lasers (QCL) opens up new attractive possibilities for the application of infra red absorption spectroscopy for plasma process monitoring and control. QCL emit in the MIR spectral range at room temperature, i.e. without the need of strong cooling. Because of that it is possible to design very compact MIR sources which are characterized by

³To whom any correspondence should be addressed.
single mode operation and high radiance power. Sensitivities in the range of ppb with time resolutions of better than ms are realizable.

With the compact and modular measurement device Q-MACS [5] it is possible to use the potential of the QCL for plasma process control or for high sensitive trace gas analysis. It combines the advantages of absorption spectroscopy in the mid infra red spectral range with the positive properties of quantum cascade lasers. In this contribution we report actual results using the Q-MACS Etch system for the in situ process monitoring of the industrial production of DRAMs. In situ measured concentration devolutions of the etch product SiF₄ demonstrate the abilities for a process control.

2. The Q-MACS Etch
On the basis of Q-MACS a two channel spectroscopical system was developed, which fulfills the special requirements for an application in a semiconductor environment. A picture of this Q-MACS Etch can be seen in figure 1 showing the separate optical module and the control unit in a rack. Beside compactness and integrability a new concept for the optical coupling to an industrial dual frequency, capacitive coupled, magnetically enhanced, reactive ion etcher (MERIE) was developed. For simple operation and avoiding of open optical pathways an optical coupling with a MIR fiber to the etch chamber has been realized. The fiber can be mounted in front of KBr windows either for side access or

Figure 1. Picture of the Q-MACS Etch with the optical module and the separate control unit in a rack.

Figure 2. Concept of the optical coupling between the Q-MACS Etch and the plasma etch chamber with the schematic of the optical board.
for top access to the chamber, see figure 2 left. In case of the side access mode the infra red beam is reflected via a mirror in the liner door and coupled back into the fiber. Because of this only one optical window is necessary. This arrangement is used for the concentration measurement of etch relevant species. In case of the top access mode, the surface of the wafer itself serves as reflective element. Because of the partial transparence of silicon and silicon dioxide in the mid infra red spectral range, this arrangement can be used for an interferometric etch rate determination.

The optical setup together with the calculated beam paths via ray tracing methods is shown in figure 2 on the right. The main components consist of the laser source, the detectors, opto-mechanical parts and an MIR fiber as well. With the help of a central beam splitter both the QCL emission is separated into a measurement and a reference channel and the input MIR radiation is decoupled from the reflected signal from the chamber. In the reference channel absorption lines of the reference gas C2H4 are measured simultaneously in order to fix the QCL emission at a distinct spectral position with the help of a control loop (line locking). Adapted to clean room requirements and for low maintenance level thermoelectric cooled detectors are installed on board.

3. Spectroscopy at the etch product SiF4

In the plasma process under study deep trench structures were etched in Si wafers using NF3 as precursor. One of the main etch products is known to be the molecule SiF4. To determine the spectral absorption characteristics for the proper selection of a QCL with its narrow spectral bandwidth, reference gas cells were filled with SiF4 and NF3 and were measured with a FTIR [11]. Figure 3a shows the absorption band of SiF4 at 1030 cm⁻¹ due to the ν3 stretch of the molecule. It becomes obvious that there is a spectral overlapping with the precursor NF3. Taking this into account, the tuning of the QCL was performed so that there is a minimum of overlapping with the NF3 absorption to minimize the errors in the concentration determination of SiF4. For the selected spectral range of 1028±0.3 cm⁻¹ the ratio between the absorption cross sections of both molecules is close to 90. The part of the absorption spectrum measured with the Q-MACS is shown in figure 3b. The laser line width of approximately 0.008 cm⁻¹ impedes to resolve spectrally the single rovibronic transitions. Using a numerical approach for the treatment of such complex spectra an effective absorption cross section of SiF4 was determined to σ=7.7(±0.7)×10⁻¹⁸ cm² molecule⁻¹ and for NF3: σ=8.7(±0.8)×10⁻²⁰ cm² molecule⁻¹ [11]. With the experimentally determined absorbance and the known absorption length the concentration of the absorbing species follows from the Lambert-Beer law. Because of the broad band absorption characteristic of SiF4 in the tuning range of the QCL it was necessary to measure the

![Figure 3a. FTIR spectra of SiF4 and NF3 from 1005 cm⁻¹ to 1050 cm⁻¹, the spectral resolution is 0.11 cm⁻¹.](image)

![Figure 3b. QCLAS spectrum of SiF4 from 1027.6 cm⁻¹ to 1028.3 cm⁻¹, instrumental broadening is 0.008 cm⁻¹.](image)
background signal for the reference signal separately.

4. Synchronization with the etch tool

The above discussed absorption behavior of SiF₄ impedes the simultaneous detection of the background intensity of the QCL during the measurement. Having a look on the time course of the plasma etch process different states in the chamber are distinguishable and can be related to different phases of the process. This can be used for the automated determination of the concentration, as illustrated exemplarily in figure 4 for a conditioning sequence. If the liner door is open the QCL radiation is no longer reflected back from the chamber and the detected signal corresponds to an offset \( I_{\text{off}} \), which is important to take into account in the calculation. As soon as the liner door is closed the background intensity \( I_0 \) is measured before the process gases start to flow in the stabilization step. During the plasma phase (ME) the signal \( I(t) \) corresponds to the actual transmission. At the end of the plasma process again it is possible to measure the background intensity \( I_0 \), which can be altered because of depositions at the window or mirror. Therefore the concentration \( N \) at a time \( t \) is given by:

\[
N \propto \int dt \ln \frac{I_0^{i} - I_{\text{off}}}{I(t) - I_{\text{off}}} 
\]

To be able to use these different meanings of the signal a synchronization between the Q-MACS Etch and the etch chamber control tool is required. This was realized by means of a communication via a customized network interface. With the exchange of attributes it was possible to interpret at any time point of the process the signal and therefore to calculate the concentration. From the changes of the background intensities \( I_0^{i} \), a degradation coefficient was determined in a linear approach and the concentration corrected correspondingly. The effect of the degradation on the concentration can be seen in figure 5 exemplarily. On the left side the signal course is shown as recorded from the measurement detector from batch processed wafers with the corresponding degradation coefficient. The sequence was measured during the etching of 25 masked wafers for deep trench structures after seasoning the chamber. Every etching process was followed by a cleaning cycle consisting of a special clean process for the cathode (ICClean) and a cleaning of the chamber (DryClean). In the right graph of figure 5 both SiF₄ concentration values are shown, the uncorrected in grey and with degradation taken into account in black. During the etching processes the differences become significant whereas the effect is less pronounced during the cleans.

![Figure 4. Time course of the measurement signal I(t) during a seasoning sequence where I_{off} is the offset, I_0^{i} and I_0^{2} the background before and after the process. The different meanings according to the actual status of the process are given: prestab, ST and ST-2 – stabilisation steps, ME – main etch, RD – ramp down and door open.](image-url)
With this the concentrations of the etch product SiF$_4$ can be transferred to the tool and used for process monitoring or control purposes. The synchronization with the etch tool fulfills therefore an important requirement for the online process monitoring with Q-MACS in typically automated high volume production environments.

5. Monitoring of the Si etch process

In the experiment reported here 25 masked Si wafers were processed for the plasma etching of deep trench structures in a batch mode. Between the etching sequences the chamber was cleaned with an ICClean and a DryClean procedure. Before the etching procedure the chamber was conditioned twice via a process-like plasma and blank Si wafers. Taken into account the above discussed degradation the resulting concentration courses were like in figure 6 presented. The individual traces were overlaid and normalized with respect to their starting time in order to compare the devolution of the SiF$_4$ concentration for each wafer. For reason of clarity all 25 graphs are split into two graphs. As already recognizable in the signal traces in figure 5 (left) the development of the SiF$_4$ concentrations during the deep trench etch processes are affected by a seasoning which is not present in the subsequent clean cycles, see figure 7. Furthermore, for the latter the SiF$_4$ concentrations were detected stable and

![Figure 5.](image1.png)

**Figure 5.** Left: course of the degradation coefficient (blue) and the measurement signal (grey) during the processing of 25 wafers. Right: the SiF$_4$ concentration values with degradation taken into account (black) and without (grey).

![Figure 6.](image2.png)

**Figure 6.** SiF$_4$ concentration devolutions during the individual etch processes. The first 13 process courses are shown on the left, the remaining on the right with same scaling. In addition the single process steps are emphasized in grey.
reproducible since the beginning of the batch. There is a time jitter in the concentration values due to
different spans between two successive processes caused by the tool control. In case of the deep trench
processes it takes up to 8 cycles before the concentration courses becomes more reproducible. This is
seen as a clear indication of chamber conditioning effects, i.e. the balancing of chemical reactions is
dominated by plasma wall interactions. In dependence of the prehistory it takes several process cycles
until stable conditions prevail in the etch chamber. Because of that the measured concentration courses
show a stronger scattering at the beginning of the experiment. Moreover, also negative values were
detected for the SiF$_4$ concentration, which is an artifact and does not have any physical meaning. The
appearance is related to an enhanced background during the process. With increasing number of
processed wafers the concentration courses become more and more reproducible as it can be seen in
the right graph in figure 6. During the etch process SiF$_4$ is built up to $6\times10^{13}$ cm$^{-3}$ which corresponds to
a partial pressure of ca. 1%. Significant different concentration values were detected for the wafer no.
22 with about 30% higher SiF$_4$ concentrations in the first fourth of the etch process. As in case of
wafer no. 6 the previous cleaning procedure failed and was stopped from the tool control. Again the
amount of detectable SiF$_4$ during the etch process depends sensitive on the chamber conditioning.

In figure 8 first results of inline data of the 25 processed wafers are presented. The etch results are
evaluated via an optical method in the infra red spectral range and statistically analyzed with the help
of a complex model. The deep trench structures are characterized besides other properties by the
trench depth and the bottom void. The latter is a measure for the area at the bottom of the trench and
for the roughness at the trench side wall. The results for the trench depth show a homogenous
distribution around 6.9µm except small deviations in case of wafer 6, 21 and 22, see left graph in
figure 8. For these wafers the trench depths are significant up to 200nm smaller. The values for the

Figure 7. Development of the SiF$_4$ concentration during the DryClean cycles.

Figure 8. Boxplot of the first inline data: deep trench etch depth (left) and bottom void (right).
bottom void show an analog distribution as the trench depth, as it can be seen on the right graph. Again the influence of the failed clean cycles becomes apparent.

6. Summary
A quantum cascade laser system Q-MACS Etch was developed for process monitoring in the semiconductor industry environment. For the first time one of the major etch products SiF$_4$ was monitored in situ in silicon plasma etch processes used for the production of DRAM modules. Based on the determination of an effective absorption cross section it was possible to measure online the molecular SiF$_4$ concentration during the process. In order to evaluate the reproducibility and the stability of the plasma processes the measurements were expanded to a batch processing of 25 silicon wafers. The required synchronization with the etch process was realized via a customized network interface. With this the Q-MACS Etch communicates with the etch tool providing the automation of data acquisition. The usage of a special MIR fiber in combination with an internal mirror is an innovative approach to ensure robustness and flexibility in an industrial production environment.

The first application of a quantum cascade laser arrangement for monitoring of industrial etch processes has opened up a challenging new option for control of demanding semiconductor production. Focused on sensitive and fast concentration measurements of molecular key components while ensuring compactness, robustness and long term stability, this new class of process control equipment has the potential to become implemented also into other fields of plasma technology.

Acknowledgements
The work for his paper was supported within the scope of technology development by the EFRE fund of the European Community and by funding of the State Saxony of the Federal Republic of Germany.

K.-D. Weltmann is gratefully acknowledged for his continuous encouragement and general support making the present work possible. The authors are especially thankful to S. Barth, M. Reinicke, S. Herberg and U. Macherius for their permanent support and stimulating climate. We are also indebted to F. Hempel, S. Glitsch and F. Weichbrodt for the technical assistance and useful discussions.

References
[1] Steinbach A, Barth S, Henke A, Wege S, Sobe J, Reinicke M and Kijowski M 2005 6-th European Advanced Equipment Control / Advanced Process Control (Dublin, Ireland)
[2] Gräßlert W, Dani I, Throl O, Hopfe V, Pietsch K, Wünsche T and Dreyer T 2006 VDI-Berichte Nr 1959 45
[3] Heason D J, Spencer A G 2003 J. Phys. D: Appl. Phys. 36 1543
[4] Sun H C, Patel V, Singh B, Ng C K and Whittaker E A 1994 Appl. Phys. Lett. 64(21) 2779
[5] Hempel F, Glitsch S, Röpcke J, Saß S and Zimmermann H 2005 Plasma Polymers and Related Materials ed M Mutlu 142
[6] Röpcke J, Glitsch S, Hempel F, Saß S, Schulz K-D, Weltmann K-D and Zimmermann H 2005 Proc. 48th Annual SVC Conf. (Denver, USA) 54
[7] Röpcke J, Lombardi G, Rousseau A and Davies P B 2006 Plasma Sources Sci. Technol. 15 148
[8] Röpcke J, Glitsch S, Hempel F, Lang N, Macherius U, Saß S, Stancu G D, Weichbrodt F, Weltmann K-D and Welzel S 2006 VDI-Berichte Nr 1959 279
[9] Welzel S, Gatilova L, Röpcke J and Rousseau A 2007 Plasma Sources Sci. Technol. 16 822
[10] Röpcke J, Welzel S, Lang N, Hempel F, Gatilova L, Guaitella O, Rousseau A and Davies P B 2008 Appl. Phys. B 92 335
[11] Stancu G D, Lang N, Röpcke J, Reinicke M, Steinbach A and Wege S 2007 Chem. Vap. Deposition 13 351