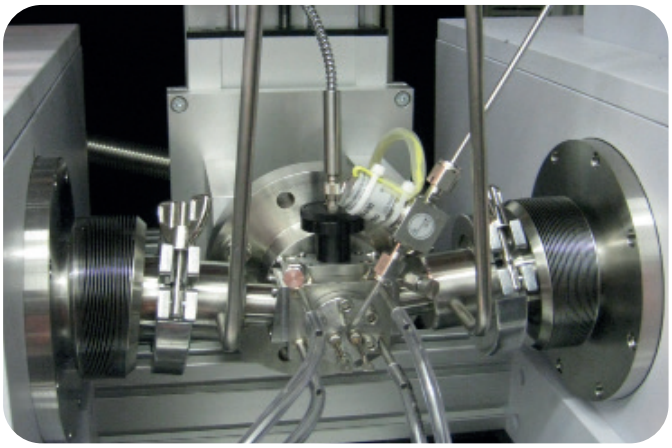

Application Note AN M96

# External Sample Chambers

Bruker Optics research FTIR spectrometers are well known for their high flexibility and usability in connection with external measurement devices. Although a large variety of external measurement modules is available (see table I) many R&D spectroscopists require specific external measurement chambers with dedicated optics layouts in order to fulfil their high demands for extremely flexible, variable and/or large size set-up. In figure 1 the Bruker Optics external chamber in its vacuum version A514/V is shown which was designed for such purposes. Typical applied accessories and measurement techniques are listed in table II.

Such types of external measurement chambers have a significant additional general value: the instrument´s sample compartment is kept free for quick standard measurements without the need to remove difficult to handle or large and heavy accessories like low temperature cryostats.

Tab. 1

**External Measurement Modules**

- XSA: standard second sample compartment A172/B
- External purge (A514/B) and vacuum chamber (A514/V)
- HTS-XT: high sample throughput accessory
- PMA 50: dedicated for variable angle PM-IRRAS and VCD spectroscopy
- RAM II: near IR FT-Raman module
- PL II: photo-luminescence module for semiconductor application
- TGA and GC/IR: hyphenated techniques
- HYPERION 1000/2000: IR microscopy
- HYPERION 3000: focal plane array (FPA) imaging
- IMAC: macroscopic imaging chamber.
- Water surface reflectance unit

## Kinematic Sample Compartment Baseplate

With the introduction of the FTIR spectrometer series VERTEX the Quick-Lock (QL) sample accessory mount became the standard for high end research spectrometers as well. QL provides easy and reproducible exchange of different types of sampling accessories without the need of any realignment. In addition the Automatic Accessory Recognition (AAR) functionality of the TENSOR II, INVENIO and VERTEX series identifies the accessory and allows automatic setting of predefined measurement parameter sets. The QL mount was introduced some years ago because the formerly used kinematic mount was not the ideal solution for routine work. There was the drawback that perfectly aligned accessories could be misaligned easily by non-experienced users.

But that kinematic sample compartment base plate - which is still used in the IFS125HR ultra high resolution FTIR spectrometer – offers the advantage that the optimum adjustment of custom made accessories is easily achievable.

In addition the optical beam height of the kinematic mount is significantly higher (92 mm) than that of the QL base plate (64 mm to 72 mm). In case the beam height of the used accessory is low enough (below 62.5 mm or 2.5 inches) a conversion into the QL-design is possible (part number S580). But for some of these accessories it is not the case. Such ones need the external measurement chamber A514/B or A514/V. With the QL-adaptor A195 which fits on the kinematic base plate upward compatibility with new QL accessories is guaranteed.

Tab. 2

**Dedicated External Accessories**

- Beam condensers
- Low temperature cryostats
- Adaptation for Quantum Cascade Lasers (QCL) emission measurements
- High pressure diamond cells
- variable temperature high pressure cells
- Black Body (BB) reference sources
- XYZ-mapping stages
- Long path gas cells
- Large diameter integrating spheres for IR and VIS spectral ranges
- Measurement units for detector characteristic determination
- Large size and/or heavy weight customer accessories
- Accessories mounted on the kinematic base plate
- Adaptation of UHV measurement chambers for in-situ analysis
- Coupling with plasma reaction chambers

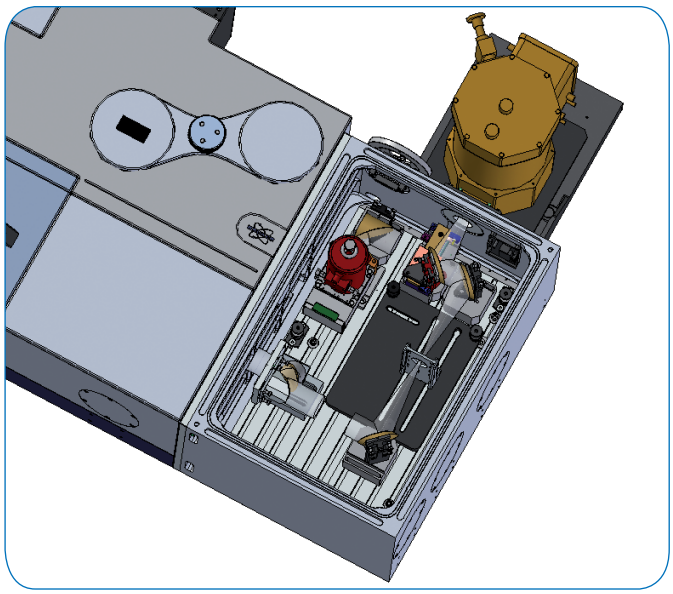

Fig 1. The image shows the external measurement chamber A514/V (vacuum version) connected to the right side exit port X1 of the VERTEX vacuum research FTIR spectrometer optics bench.

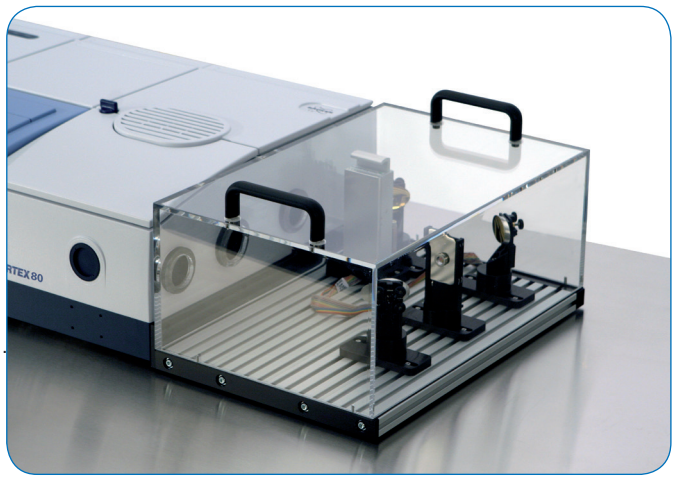

Fig. 2: The photograph shows the purgeable external sample chamber A514/B with transparent cover, transmittance sample holder and detector mount equipped with room temperature DTGS-Detector connected to the VERTEX 80 at its right side exit beam port.

## Description of the A514/V and A514/B

The external chamber A514/x provides not only sufficient beam height but in addition a large space (ca. 320 mm x 580 mm) which allows it to be used as a work bench for variable and sophisticated optics layouts. It accepts internally the standard DigiTect room temperature as well as a liquid N2 cooled detector (like MCT's) and externally the liquid He cooled bolometer detector. Dedicated layouts have been already configured, examples are shown in figure 3. Adapted on the left side of the VERTEX optics bench it might be used as an additional detector chamber which will allow the automatic switching of 3 permanently mounted detectors or even more. This could be particular of interest in case the complete spectral range from the far IR/THz (e.g. 5 $cm^{-1}$) through the mid and near IR up to the VIS and UV spectral ranges (e.g. 50,000 $cm^{-1}$) should be applicable. In connection with the new beamsplitter exchange unit BMS-c (T650/BV) this is even possible under vacuum conditions in a fully automated way. Typical measurement examples are demonstrated in the Bruker Optics product note "T650-BV".

In connection with the flexible INVENIO R and VERTEX series different chamber positions and orientations are possible which are at the right, left and front side of the spectrometer optics (see figure 3). Additionally dedicated application chambers can be offered which for example allow modulated Photo-Luminescence (PL), Photo-Transmit-

tance (PT) and Photo-Reflection (PR) measurements even in connection with a low temperature cryostat. The optics are equipped with moveable mirrors with the goal that the sample does not need to be moved and the optics need not to be vented to switch to the different measurement techniques. The optics layout is shown in figure 4. Customers in semiconductor physics research institutes are using it successfully and have published outstanding measurement results [1] and [2].

## UHV Chamber Adaptation

Quite often the demand arises to adapt to the FTIR spectrometer optics a large gas phase measurement cell, an Ultra High Vacuum (UHV) chamber or a Plasma reaction chamber. Due to their special dimensions a complex optics design is required to fulfil the required energy radiation throughput and measurement sensitivity. Besides the directly attached vacuum chamber an additional and separate detector chamber is usually added.

Application areas for such designs are for example the analysis of Self Assembled Mono-Molecular (SAM) and even sub-mono-molecular layers, catalytic surface reaction studies, Chemical Vapour Deposition (CVD) analysis or atomic layer measurements. Mono-molecular layer analysis is typically made under grazing angle IR reflectance (GIR) using polarised light in the mid IR spectral range with high sensitivity liquid $N_2$ cooled MCT detectors. Transmittance set-ups are used as well and in single cases LHe cooled far IR bolometer detectors are connected additionally. The typically used set-up is the part number W109/UHV which is shown in figure 5 without a UHV system.

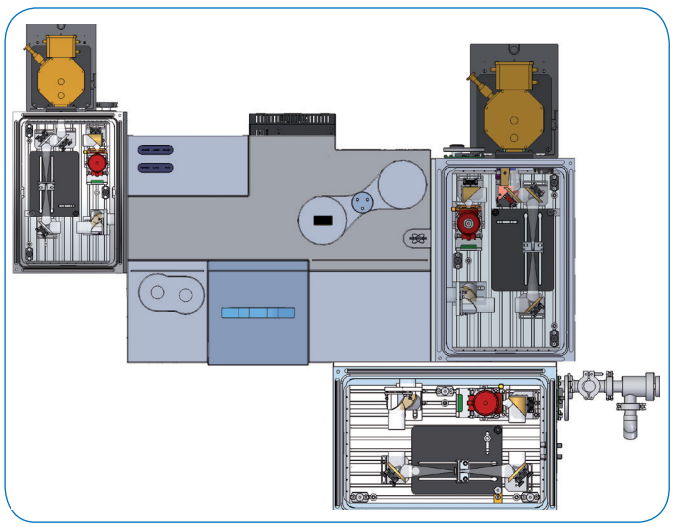

Fig. 3 The image shows the orientation possibilities for the external measurement chamber A514/V with externally adapted bolometers at the right and left side and without a bolometer at the front side of the VERTEX spectrometer optics bench. All 3 chambers could be mounted simultaneously if required.

Depending on the sample size, the geometric boundaries of the UHV chamber and the available optics ports, the optics layout needs to be optimized for each particular case. As an example for grazing angle measurements an optimized slit aperture for highest energy throughput and smallest sample sizes is available (W109/ATP).

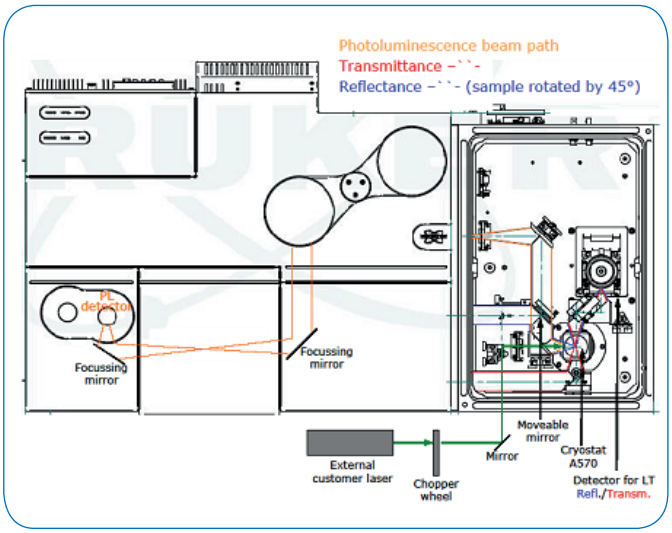


Fig. 4 Optics layout of the dedicated and automated PL-PT-PR measurement vacuum set-up chamber typically used in connection with the VERTEX 80v vacuum FTIR spectrometer for semiconductor material research and development [2]

The UHV supply of the UHV measurement chamber and its vacuum interface is provided by the user (see fig. 7). For sure the Bruker Optics application and technical staff will provide necessary support and will share their know-how with the user. Bruker has installed adaptions for different UHV System provided by various companies which might be contacted by the customer directly to ask for UHV support.
A dedicated adapted solution supplied for an already existing UHV system is shown in fig.6.

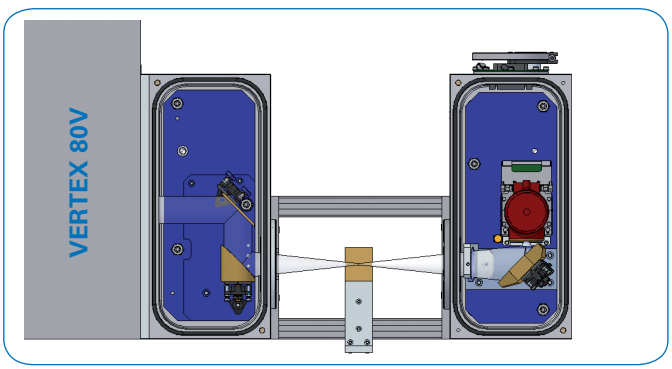


Fig. 5: The external UHV adaptation- and detector chamber W109/UHV is shown in the typically supplied configuration in order to allow performance test prior to the customer supplied UHV chamber being adapted.

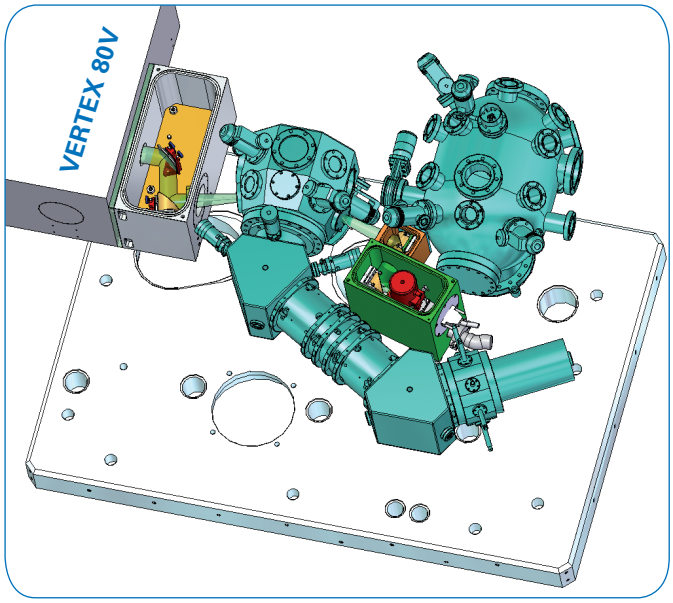


Fig. 6: VERTEX 80v adapted to a customer UHV chamber. The detector chamber was specially designed to fit into the existing specific UHV system.

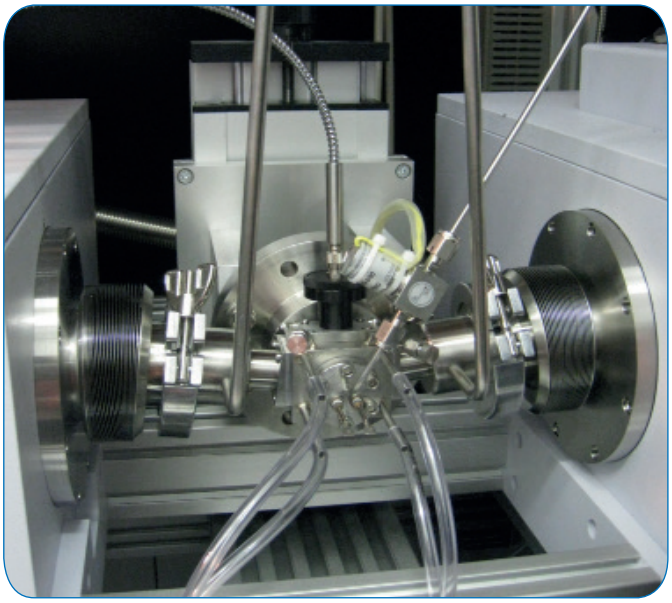

Fig. 7: Photograph of a customer supplied UHV sample chamber, vacuum tight interfaces for the adaptation and detector chambers as well as required supply lines.
With courtesy of Dr. Mathias Laurin, Universität Erlangen, Germany

## Summary

With the external purge and vacuum chambers an interesting add-on to the flexible INVENIO R and VERTEX FTIR spectrometers series for demanding and specialized research applications is available. The chambers allow the use of the previously used kinematic sampling accessory and non-standard optical set-ups for which the spectrometer sample compartment is not applicable. For the adaptation of UHV systems the use of an additional detector chamber can be offered. External measurement chambers are successfully used in different research areas which are reflected in the significant number of scientific publications for which in particular Bruker Optics vacuum FTIR spectrometers have been applied.

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