ABSTRACT

The ESA Materials Space Evaluation & Radiation Effects laboratory has designed and developed a new outgassing facility for high temperature, DOK (Dynamic Outgassing Knudsen). The purpose of DOK is to measure material outgassing to be used in dynamic outgassing models for predicting the evolution of molecular contaminants for missions in extreme environment, e.g. Bepi Colombo. DOK is based around a Knudsen cell and four Quartz Crystal Microbalances (QCM) mounted inside a vacuum chamber. The Knudsen cell and the associated temperature control have been designed to reach very high temperature for outgassing, i.e. in the order of 400 °C.

The total outgassing rate from the material sample, loaded in the Knudsen cell, is determined by measuring the deposition rate of material ejected from the orifice of the Knudsen cell onto a QCM cooled at liquid nitrogen. The rate of condensable matters at up to three different temperatures is directly measured on the other three QCMs.

A computer based data acquisition system controls the Knudsen cell temperature and QCM parameters and acquires the test data.

DOK is designed to run both the ESA VBQC and the ASTM E 1559 standard test.

1. INTRODUCTION

With the coming space missions operating in high temperature environment, like Bepi Colombo and Solar Orbiter, it is necessary to characterise the outgassing behaviour of the selected material at high temperature to ensure that they maintain structural and chemical integrity when exposed to the mission conditions. Bepi Colombo and Solar Orbiter are missions that require orbits very close to the Sun, with solar irradiation on the order of 10 solar constants. This implies an extreme thermal environment requiring specialised high temperature, high radiation resistant materials. Therefore, dedicated verification equipment is required for testing the outgassing behaviour and predicts the evolution of molecular contaminants of candidate materials. The Total Mass Loss (TML) and the Collected Volatile Condensable Material (CVCM) of materials with the desirable characteristics have been measured up to 125°C by various European organisations and companies including ESA-ESTEC, ONERA Toulouse, CNES Toulouse, Austrian Research Centres (ARC). However, none of them is able to qualify a material for space application at high temperature.

In response to the need for dynamic outgassing test at high temperature, the ESA Materials Space Evaluation & Radiation Effects (TEC-QEM) laboratory developed the DOK (Dynamic Outgassing Knudsen) facility, unique in its kind in Europe. DOK is the first European facility which fulfils strategic needs for these European high temperature space missions.

2. DESCRIPTION OF THE FACILITY

The DOK facility is located in the TEC-QEM laboratory at ESA-ESTEC, in Noordwijk, The Netherlands. DOK was designed according to ASTM E-1559 to fulfil both the ESA VBQC and the ASTM E-1559 standard tests.

The facility consists of a vacuum chamber, a vacuum interlock chamber, a temperature control system, an effusion cell (Knudsen cell), four Quartz Crystal Microbalances (QCM’s), a Residual Gas Analysis (RGA) and a data acquisition system. Figure 1 shows a picture of the DOK apparatus. Two QCMs are mounted at 20° and two at 10 ° with respect to the normal of the cell orifice.

Figure 1: DOK facility apparatus
The sample is placed in the Knudsen cell via the interlock chamber and then introduced into the main chamber at a pre-defined distance and angle with respect to the QCM surfaces. The sample is then heated up in high vacuum by a controlled heater. The outgassing flux leaving the cell orifice impinges on four QCMs which are arranged to view the orifice. The molecules not directed towards the QCMs are trapped by a cryogenic shroud, cooled with a cryogenic heat sink. Figure 2 shows the schematic of the DOK.

One QCM is operating at cryogenic temperature for measuring the total outgassing rate. This QCM is installed on a cryo plate cooled down via liquid Nitrogen. The other three QCMs are operating at defined temperatures, e.g. -25°C, -50°C and -75°C respectively, for the measurement of the deposition of outgassing condensable. These are installed on a plate thermally decoupled from the QCM cryo plate and cooled down by a cryo-bath. The RGA is used to identify the gases present in the vacuum during the execution of the test. Three PT100s monitor the temperature of the cryo-shroud, three others the Knudsen cell, one the QCM cryo-plate and one the QCM cold plate.

The design of the Knudsen cell is a critical element for the success of the test. It was designed to ensure that the temperature is uniformly distributed throughout the inner cavity, where evaporation takes place and the plate of the orifice. Dedicated thermal insulators were placed to decouple the Knudsen cell from the cryo-shroud. An additional shield has been mounted to limit the view of the effusion cell to the cryo-shroud.

3. TEST RESULTS

Dynamic outgassing tests were performed in the DOK according to the ESA VBQC standard test method. The sample was heated up inside the Knudsen cell from 25°C to 125°C in steps of 25°C every 24 hours in high vacuum, i.e.1x10^-7 (+2x10^-8/-2x10^-8) mbar. The QCM, on the QCM cryo plate, remained at an average temperature of -177°C. Data recorded from this QCM were post-processed to calculate the TML, shown in Figure 3.

The other three QCMs, on the QCM cold plate, were operating at -25°C, -50°C and -75°C respectively, for the measurement of the CVCM, shown in Figure 4. The shroud was maintained at -170°C to trap those molecules not directed towards the QCMs.

![Figure 2: Schematic of DOK](image)

![Figure 3: TML obtained from the post-processing of the dynamic outgassing data](image)
The TML and the CVCM in units of percent of initial sample mass are calculated according to Eq. 1 and Eq. 2 respectively:

\[
TML = 100 \times \left( \frac{F_1 K_s [f(T_q, T_s, t) - f(T_q, T_s, 0)]}{m_s(i)} \right) 
\]

\[
CVCM = 100 \times \left( \frac{F_q K_s [f(T_q, T_s, t) - f(T_q, T_s, 0)]}{m_s(i)} \right) 
\]

Where:
- \( F_1 = 490 \text{ cm}^2 \), view factor of the cryo QCM to the effusion cell orifice as calculated according to [2], [3], [4], [5].
- \( F_q = 490 \text{ cm}^2 \) or \( 441 \text{ cm}^2 \), view factor of the QCMs to the effusion cell orifice as calculated according to [2], [3], [4], [5].
- \( K_s = 1.96 \times 10^{-9} \text{ (g cm}^{-2}\text{ Hz}^{-1}) \), QCMs mass sensitivity factor
- \( f(T_q, T_s, t) \) = QCM frequency at time \( t \) (Hz)
- \( f(T_q, T_s, 0) \) = QCM frequency at time 0 (Hz)
- \( m_s(i) \) = sample mass (g)

Other tests were performed to verify the capability of DOK to reach high temperature, i.e. 450°C, in the Knudsen cell and keep the shroud and the cryo QCM at cryogenic temperature.

Figure 6 shows that the TEC-QEM DOK facility can be used for dynamic outgassing tests that involve high temperature samples. This capability is of direct benefit to space missions that operate in extreme thermal environments.

Figure 5: TML long term prediction obtained from the mathematical treatment of the dynamic outgassing data

Figure 6: Knudsen cell and shroud temperature graph during DOK high temperature validation test
4. CONCLUSION

A new facility, unique in Europe, has been designed and manufactured in the ESA-ESTEC TEC-QEM laboratory to perform dynamic outgassing tests at high temperatures. Recent validation tests showed that the DOK is capable of heating a sample up to xx °C whilst also keeping its cryo shroud at stable cryogenic temperature.

Up to now the facility has successfully run dynamic outgassing tests up to 125°C but it has been demonstrated to operate at much higher temperature for the characterisation of spacecraft materials for use in future space missions to extreme thermal environments.

5. REFERENCES


