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BURSTING TEST OF A SILICON CARBIDE MICRO REACTOR

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This document describes and presents the results of a pneumatic bursting test of a CORNING AFR® G4 industrial silicon carbon reactor performed at INERIS in France. The reactor burst beyond 150 bar, well above its design pressure of 18 bar. The kinetic energy of fragments ejected during the bursting and the pressure effects were measured. The observed effects are below the minimum levels of significant injury for human body.

1. Introduction

The micro reactor technology has notably gained more and more interest over the past years, and its penetration in production plants is increasing rapidly. Micro reactors are devices operating at flow rates up to several thousands of millilitres per minute, with a characteristic dimension (diameter) ranging from several micro to several millimetres (the latter being sometimes named “milli “or “meso” reactors) (Jensen, 2017).
This kind of equipment offers a much better heat transfer than conventional equipment; the higher heat release management allowing to operate at a higher temperature, which leads to faster kinetics. They also provide a better mass transfer that can increase the reaction rate. Therefore, they allow producing similar throughput as batch processes with a much lower quantity processed at the same time, leading to a drastic reduction of damages resulting from a failure or a chemical runaway. In that respect, micro reactor technology is clearly part of IST (Inherently Safer Technology).
However, operating at higher temperatures means operating at more severe condition. A sudden shut down of the feeding pump or cooling circulation may cause a temperature increase and the formation of hot spots leading to a reaction runaway accompanied by gas or vapor production. A micro reactor being generally full of liquid (except for gas-liquid reactions), a local pressure increase would not be damped by a free volume as in a batch reactor, and the pressure could rise very rapidly into the reactor. The rate of pressure rise would depend on several factors, such as the capacity of the material constituting the reactor wall to partially absorb the pressure wave (elasticity), the head loss in the reactor and the rate of gas generation by the reaction. As the pressure would rise very rapidly, a conventional mechanical protection device (PSV) should not operate as fast as requested due to its mechanical inertia. Bursting disk could be suitable providing that it would be installed precisely at the point where the pressure increase would be initiated. Ultimately, the micro-reactor itself would acts as a bursting disk.
This phenomenon so far has not been studied from a theoretical point of view, and modelling may not be so relevant as many parameters are involved.
End users may therefore question about the effects of a pressure rise resulting from a reaction runaway in a micro reactor, and what could be the consequences on the direct surrounding of the reactor, in particular on workers.
In order to determine the consequences of a rapid pressure rise in a micro reactor, a pneumatic bursting test was carried out at INERIS on a CORNING® Advanced Flow™ G4 industrial reactor.

2. CORNING® Advanced Flow™ Reactor (AFR)

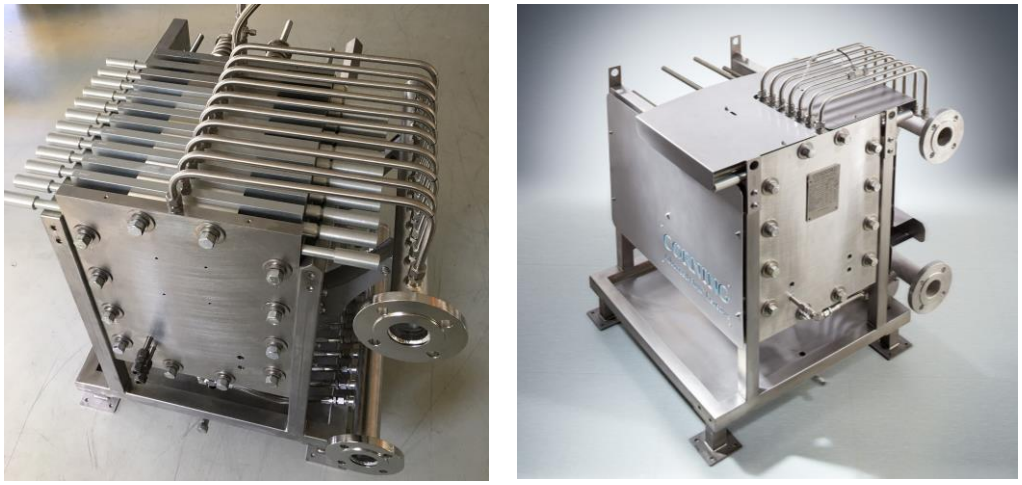
Corning is involved in micro-reactor development and sales since 2002. The first reactors were made of glass and dedicated mainly to lab development and production for high added value products (pharmaceutical industry). High throughput micro reactors for industrial production (several thousands of tons per year) have been introduced on the market for more than seven years, and several industrial units are in operation in Europe, India and China.

In the micro reactor for industrial production (G4 AFR), the process flow is contained in plates made of sintered silicon carbide (SiC). This material has very good thermal properties, is not subject to thermal shock and has a large thermal inertia, another key point for safety in case of sudden shut-down of the cooling system. On top of that, it has been chosen because it is virtually not subject to corrosion by any medium (including all acids, caustic and hydrofluoric acid) at the design temperature (ranging from -40 to +200°C).

The G4 AFR looks like a plate and frame heat exchanger with a stack of SiC plate for process, interposed between metal plates for heating or cooling medium.

In order to protect people against any unexpected liquid projection or bursting, it is surrounded by a stainless-steel casing (see picture 1).

Even though G4 AFR has a small volume (typically a few liters), it is considered as a pressure vessel and complies with European and international regulation (PED in Europe, SELO in China, KGS in Korea….).



Picture 1. G4 AFR with heat exchange piping without stainless-steel protective casing (a), G4 AFR with protective casing (b).

3. Bursting test and results

The bursting test of a G4 AFR model was performed at INERIS, the French National Institute for Industrial Environment and Risks. The mission of the institute is to evaluate and prevent accidental or chronic risks to human and the environment related to industrial installations, chemical substances and underground operations. INERIS has experimental facilities for conducting large-scale tests.

3.1 Preliminary study

A predictive analysis of the maximum potential effects of the bursting was conducted before the test to determine safety distances for operators. The pressure effects and the projection distance of the fragments were calculated using the PROJEX EPHEDRA software (Heudier, 2015). The maximum projection distance was estimated at 30m from the reactor and an overpressure of 50 mbar was estimated at 5m (see figure 1).

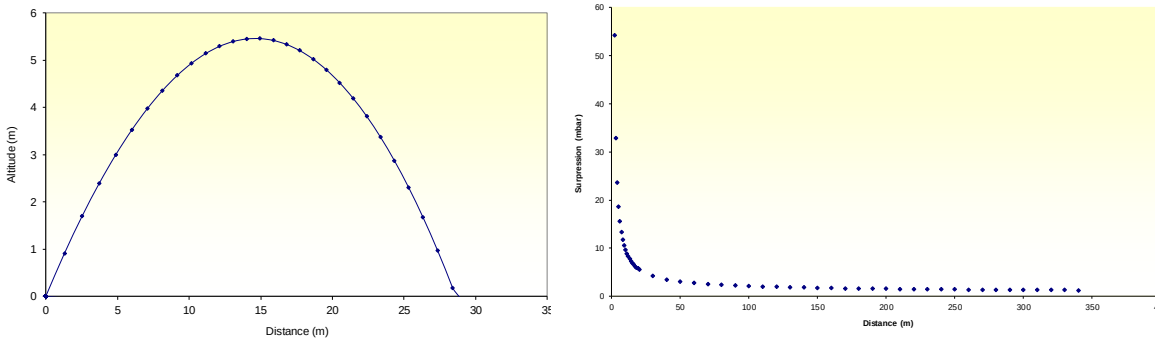
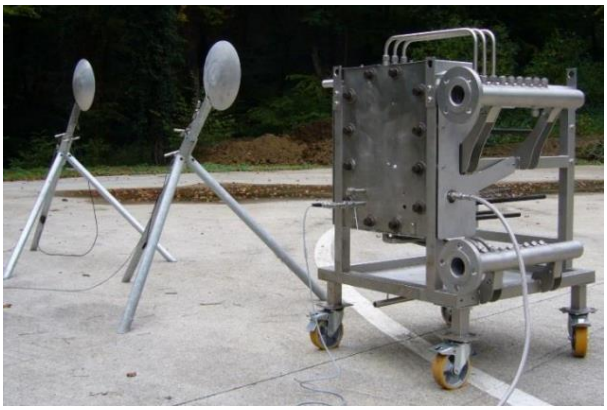


Figure 1. Maximum projection distance (a) and overpressure (b) as a function of distance to the reactor calculated with PROJEX EPHEDRA software.

3.2 Test process

The reactor model tested was made of two SiC plates interposed between three metal plates surrounded by the stainless-steel casing. The test consisted in causing the rupture of one of the SiC plates by rapidly introducing compressed air from a pressurized tank to simulate a quick pressure increase in the reactor. The test was conducted by carrying out several successive sudden loads of the plate until bursting, using remotely controlled pneumatic valves.
In order to record information on overpressure and projection of fragments that could be produced by the bursting, the following instruments were installed on or close to the reactor (see picture 2):

- A piezoresistive pressure transducer on the outlet of the pressurized SiC plate,
- Two piezoresistive pressure transducers placed on lenticular supports and positioned respectively at 1m and 2.5m from the center of the reactor, in the lateral axis of the plates,
- A high-speed camera (1000 pictures/sec), also placed in the lateral axis of the plates,
- In addition, two conventional cameras were also placed to monitor the test.



Picture 2: Test installation with atmospheric pressure sensors and pressure sensor on the SiC plate (middle of the picture). The cameras are not visible on the picture.

3.3 Test results

The pneumatic rupture of the plate occurred beyond 150 bar, well above the design pressure of the reactor (18 bar).

Reactor structure damages

After the bursting, the global structure of the reactor did not show significant damages (see picture 3).
The two SiC plates were broken during the rupture and the two heat transfer fluid plates surrounding the pressurized plate were disengaged. In addition, the lower part of the protective casing was partly torn off (see picture 4).



Picture 3. AFR before test (a) and after test (b).



Picture 4. The protective casing was partially torn off.

Pressure effects

The release of the pressure contained in an enclosure during its pneumatic rupture results in the external propagation of an atmospheric pressure wave.
The pressure recorded close to the reactor during the bursting did not show a significant pressure increase. The peak of overpressure measured at 1m from the center of the reactor was 6 mbar, and 2 mbar at 2.5m (see figure 2). It is also to notice that the pressure release in the SiC plate after breakage lasted 15 milliseconds.

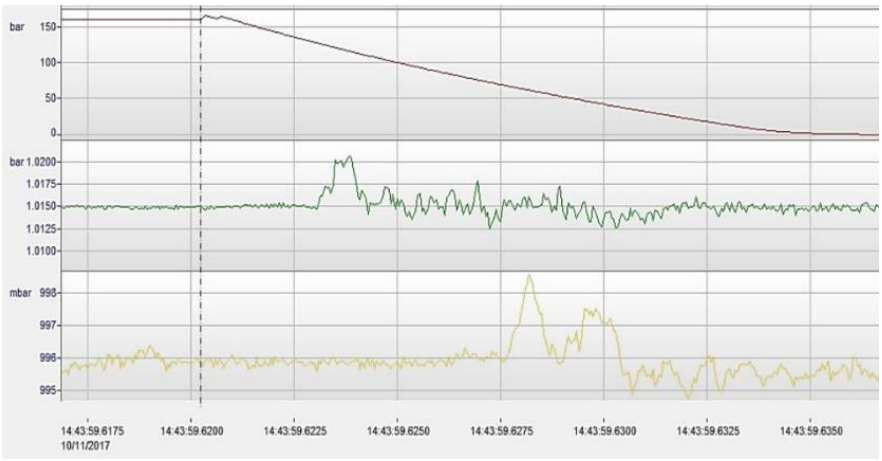


Figure 2: Pressure evolution after bursting: top dark line represents the pressure evolution inside the pressurized SiC plate, the green line is the atmospheric pressure measured at 1m from the centre of the reactor, the yellow line is the pressure measured at 2.5 m from the centre of the reactor.

Projection effects

24 small SiC pieces were projected by the bursting around the reactor through little openings of the casing (see picture 5). Their weight was less than 25 grams, and the maximum distance of projection was 6 meters.

The projection distances and the intensity of the pressure wave observed during the test are well below the estimates of the preliminary study. It is likely that the presence of the casing and the formation of multiple cracks in the SiC plate rolled the flow of pressurized air, thus limiting the intensity of the pressure effects. The projection effects were also limited by the protective casing which prevented the ejection of a number of plate fragments outside the reactor structure and by the fixing of the plates to the chassis.

3.4 Estimation of impacts on people and environment

Estimation of potential consequences of projection effects

A missile is likely to produce two types of impacts on human or on structures: shocks, that can cause fractures in the human body or lead to the deformation of structures, and penetration, which is mainly the case for small fragments. The consequences of the impact of projectiles formed by the bursting of the plate were estimated by calculating their kinetic energy and comparing it to the thresholds related to the effects of penetration of small fragments (less than 1 kg) defined in the French pyrotechnic regulation (FR, 2007).

The kinetic energy of the projectiles was calculated from their velocity assessed from the recording of the high-speed camera (see figure 3). The maximum kinetic energy calculated was less than 1 Joule, far less than the threshold of occurrence of significant injuries (8 J) and damages on structures ($3{,}7.10^5$ J).

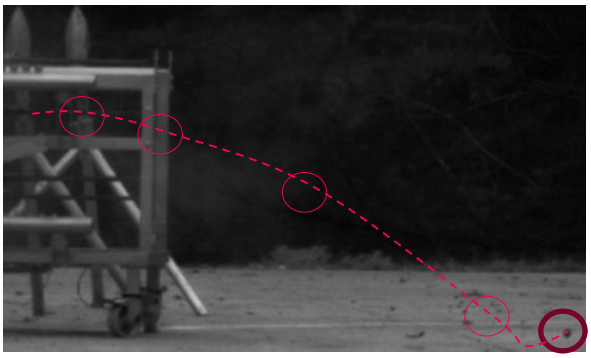


Figure 3. Extracts from the recording of the high-speed camera showing the path of a SiC fragment that allowed the estimation of the kinetic energy of projection.

Estimation of potential consequences of pressure effects

A pressure wave is likely to induce bending or shear forces in the structures and compression in the human body. A pressure wave can also propel projectiles or cause the fall of an individual. In order to estimate the potential consequence of pressure effects of the bursting, the intensity of the pressure wave measured around the reactor was compared to the thresholds of pressure effects on the structures and on the human body proposed in the Decree of 29 September 2005 relative to the assessment of dangerous phenomena that can occur in Classified Installations (FR, 2005). The intensity of the pressure wave observed after the pneumatic rupture (6 mbar) is well below the threshold of appearance of effects on individuals and structures (20 mbar).

4. Conclusion

The burst test performed at INERIS on a Corning G4 micro reactor partially made of silicon carbide has shown that under test conditions, it was unlikely that any people, even staying close to the reactor, would have been significantly injured. In addition, consequences for the close environment would be very limited.
The bursting pressure (beyond 150 bar) is significantly higher than the design pressure (18 bar) which constitute a high safety margin in case of mis-operation.
The mechanism of rupture of SiC leads to a multiple opening of the SiC plate, which limits the effect of pressure and kinetic energy of fragments.
The mechanical design of the reactor, as well as the stainless-steel casing have also a positive impact in the limitation of the consequences of the bursting.
The consequences both in term of pressure wave and projection of fragments are significantly lower than the potential maximum effect calculated by the PROJECT EPHEDRA software.
Although the test was conducted with compressed air, it gives a good trend of what could happen after a severe chemical reaction runaway leading to a rapid pressure increase by gas or vapor generation.

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