

# SINEO Microwave Digestion Core Technology (2012)

## I . Technology of Vessel:

### 1. Composite fiber outer vessel

In recent years, the application of composite fibers is becoming increasingly popular, and it is reported that 40% of the fuselage cover of Boeing 787 and Airbus 380 is made up of the composite fibers since such material has better hardness and rigidity as well as a little elasticity and it is light and thin as well. Besides, a lot bumpers in car bodies also apply such materials. Therefore, it is an ideal material which may substitute metal. When choosing material for microwave digestion high pressure reaction outer vessel, the manufacturers has tried polysulfone, ceramics and PEEK (Polyetheretherketone), but not so ideal, and finally, the composite fibers come to the mind of CEM and SINEO. The out vessel manufactured by CEM is made from composite fibers while covering PFA and leaves its two ends open, therefore, it may be corroded by acid gases easily and then the materials adjacent to the two ends may be loosen and deformed, thus causing the hardness decrease and the inner vessel leak air or even explode. As for this problem, SINEO applies processes of complete covering of PFA and spraying coating of PFA to wrap the composite fibers up, thus offering better corrosion protections for it.



CEM outer vessel shows a bell mouth with time growing



PEEK out vessel

The pressure-withstanding capacity of composite fibers is better than that of PEEK, and PEEK, due to its poor acid resistance, may make the inwall looser and its hardness poorer when the acid mist penetrating to certain depth of the inwall during the application, and then it may be broken into pieces under high pressure working. However, the composite fibers, woven in high density, may not be torn easily. Even in high pressure destructive test, the outer vessel may be still in good condition under pressure of 20MPa (200 atm) or higher. Hence, it may be called indestructive high strength composite materials.



SINEO composite fiber outer vessel covering with PFA completely



Composite fiber vessel spraying with PFA completely

## 2. Automatic decompression explosive safety plate

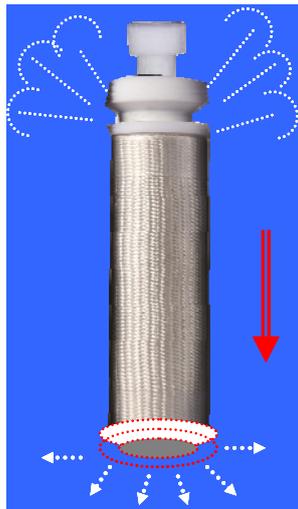
Those foreign products usually apply explosion-proof breaking decompression technology for passive safety protection, but the instantaneous strength caused by the chemical reaction could hardly be estimated and the explosive force incurred by improper operation may not be released rapidly from the small gas holes. Consequently, a lot manufacturers now tend to apply automatic release valves, such as non-metal spring cap, to let the air and pressure escape automatically after over pressure. But the vessel cover and non-metal spring cap may be deformed due to frequent escaping of air after over pressure and cannot be restored; and a lot volatile elements will escape along with it, so the tested recovery rate may be lowered as well.



Automatic release valves are deformed and broken after several escaping and cannot be restored.

To cope with this problem, SINEO develops the (patent) technology of decompression explosive safety plate. The outer vessel may withstand high pressure due to the application of composite fiber materials with high strength, and vertically, it may use this plate (piece) to release the pressure by automatically lifting the cover since its structural design may allow automatic fracture when the pressure reaching a certain level. The digester is perfectly sealed under normal operation, and only when the pressure inside the vessel reaching a certain level which may pose a threat to safety, the explosive plate will be broken to release the pressure. To a certain extent, it may make the unmanageable vertical explosive force manageable for avoidance of unnecessary dangers

caused by such force.

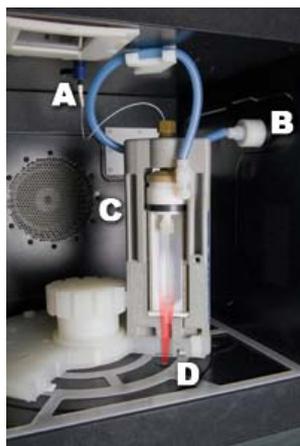


Functional scheme of decompression explosive safety plate

## II. Unique pressure-measuring technology

### 1. Piezoelectric crystal pressuring-measuring technology

At present, most manufacturers mainly apply air-pipe to measure and control the pressure inside the vessel, however, the air-pipe, since quite long, usually 400-600m, may be twisted or broken easily. Moreover, it has high requirement for sealing as the sample of master vessel may have cross contamination, and the pressure shall not be too high, otherwise, the air-pipe will break. And the digestion rotary table shall rotate 360 degrees back and forth to prevent the air-pipe being twisted.



While the air-pipe measuring pressure, the rotary table shall rotate 360 degrees back and forth to prevent it being twisted.

As for this problem, SINEO, after years of research and development, acquires the technology of measuring pressure by piezoelectric crystal inside the chamber. It, with high reliability and durability, may measure the pressure not higher than 12MPa. The measurement cable is short, only 120-140mm, so it cannot be twisted or broken, and there is no sealing joint, therefore, no air leakage, quite easy to operate. Its greatest advantage is that it may isolate the pressure element and the sample completely, leaving no chance for cross contamination.



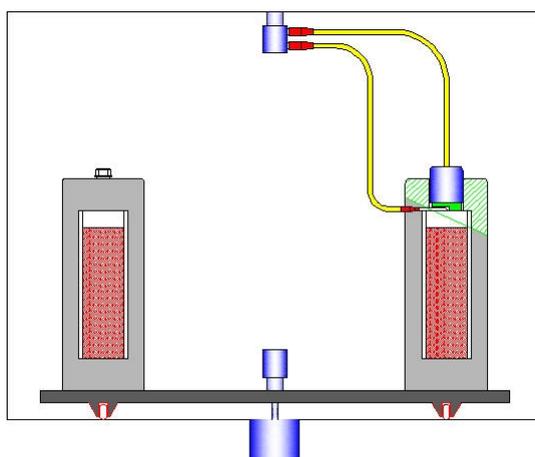
The piezoelectric crystal, like electronic balance, is installed at the bottom or at the top of the vessel and delivers pressure parameter via electrical signal.

2. Measurement device and digester rotate with rotary table synchronously in the same direction:  
The microwave digestion device used at present, if connected by pipelines, requires the rotary

table to rotate 360 degrees (except the Anton Paar wireless transmission signal or CEM-40 vessel which may measure the temperature by infrared at bottom but cannot measure the pressure.). It's defects are: the temperature-pressure measurement pipes are long and may be twisted or broken easily as it shall change directions over and over again with the rotary table. There will be a standstill area for change of direction for each circle, so the digester may not absorb microwaves evenly/ Moreover, the motor of the rotary table, since having to stop frequently, may be easily damaged, thus having a short service life.

The measurement connection device developed by SINEO may have the temperature-measurement cable rotate synchronously with the rotary table of digester, and it has the following advantages:

1. The temperature-pressure cable is short, only 140-200mm, and cannot be twisted, therefore, it may operate smoothly.
2. The rotary table may rotate to one direction continuously, allowing even microwave heating.
3. The motor of rotary table, since having light load, may not be damaged easily and have a long service life.
4. The structure is quite simple and may be assembled or disassembled easily, and the depth cooling of digester, as it may be moved out of the chamber, may improve the turnover utilization rate of chamber.



**The electrical pressure and temperature signals may be delivered to the computer outside the chamber via a patent junction box, and the junction box may allow the digester rotate to one direction continuously inside the chamber.**

### III. High throughput integrated vessel frame with over 40 vessels

With the increasingly higher demands on the number of inspection samples, high throughput multi-samples simultaneous digester becomes a new trend for development. However, those manufacturers' product we used has poor pressure-withstanding capacity, and the main problem is the unreasonable design of the frame structure,

1. The vessel frame can only place and position the digesters and cannot support the pressures of them, therefore, the digesters have poor pressure-withstanding capacity and can only work under 2MPa and 210°C;
2. Since the digesters may not have vertical support for pressures, the thread at the mouth of the dissolving cup may be easily broken;
3. The safety protection of digesters may release pressures via the deformation of vessel covers, but the covers are made from nonmetal materials and may not easily restore after deformation under the working of temperature and pressure, therefore, they may easily lose elasticity after several times and lose the sealing function as well. This is why there are decreasing samples and dried samples inside the chambers.



Anton Paar 48-vessel rotor



CEM 40-vessel rotor



Milestone 41-vessel rotor

For those problems, SINEO develops a new high throughput vessel frame under the following design principle:

1. The frame is connected by several new high-strength alloy draw bars, having considerable supports for pressures ( $\geq 10000\text{Kg}$ ), and each digester may enjoy full support from the frame at its top and bottom and may ensure the digester free from deformation and leakage under pressure of 4MPa and temperature of 250°C.
2. This design may allow the microwave field stirs inside the chamber with the rotation of frame, enabling the 40-70 high throughput digestion vessels being evenly heated in the microwave field.
3. The frame is designed with safety decompression explosive plate and may realize vertical quantitative pressure release by blowing out, ensure the safety operation of it.
4. The frame may be designed to allow the digester to realize double control of temperature and pressure.



SINEO 40-vessel high throughput rotor supported by high intensity alloy

#### IV. Light and flexible trolley

The high throughput digesters and their rotary tables (rotors) all include 40 or more digestion vessels. When all the vessels are filled with samples and solvents, its total weight may reach 15-20kg, and since most people working in the lab are women, such weight may be hard for them to put it in and take it out of the chamber.



Anton Paar 48-vessel rotator



CEM 40-vessel rotator

SINEO develops a light and flexible trolley as for this, and it may put the high throughput digestion rotary table in the chamber and take it out easily, reducing the working intensity of operators, preventing the operator touching the high-temperature and high-pressure digester directly and avoiding being damaged by acid mist, thus ensuring the safety of operation. Such trolley may move the digesters to any place of the lab flexibly, convenient for weighing, reaching a constant volume and other operations. Other electric tools may also apply to the operation of vessels for convenient sealing and opening.

