# CURRENT STATUS OF ENAMELED MEDIUM–PRESSURE REACTOR

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# Current Status of Enameled Medium-pressure Reactor

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**Abstract** This paper mainly discusses some special requirements in the design of medium-pressure enameled reactor, customary practices in the process of manufacture and inspection, and key points and applications of quality management.

**Key words** enamel glass, medium-pressure reactor,design, manufacture, inspections, quality management, application

The national standard of enamel glass (pressure vessel) has evolved from (1965 edition), 1969 edition, 1979 edition, 1992 edition to 2003 edition. There are 73 item standards about the products. The inspection standard of finished products, titled “Technical Conditions of Enameled Equipments” with standard No.HG2432 has also developed from 1979 edition, (1987 edition,) 1993 edition to 2001 edition and its latest edition is under establishment. The enamel pressure vessels are classed, according to their functions, into enamel stir vessel (detached type and closed type), enamel storage vessel (detached type, closed type and horizontal type) and enamel distillation vessel (detached type and closed type). In addition, there are enamel heat exchanger (disk type, bushed type and tube bank type), enamel column and enamel dryer etc. Enamel pressure vessels range from 300 to 3600 in diameter, from 50l to 80000l in volume. They have nominal pressure of 0.25MPa, 0.6MPa and 1.0Mpa (according to under cylinder’s pressure, the same below) and working temperature from -20℃ to 200℃. The standard doesn’t cover all specifications of products, e.g. up to 20000l for enamel stir vessel in current standard and from 50l to 40000l in new standard under consideration.

The manufacture of enamel medium-pressure reactor began from 1980’s because the process media made enamel reactors necessary. In jiangsu and zhejiang, our market research has found that enamel medium-pressure reactors are mostly used in the production of agricultural medicine and fine chemical, and scientific research. Our company is able to design enamel medium-pressure reactor up to 10000l in volume so far and up to 20000l later, including disign pressure 1.6MPa and 2.5MPa series.Temperature range is 0℃~200℃. 1.6MPa series takes priority due to market demand.The design pressure in the jacket is 1.0MPa and the design temperature is 180℃.

Our company has made a research into the practicability of design technology of enamel medium-pressure reactor according to market demand. After preliminary designs, design software SW6-98 (The design and strength calculations are based on standard No.GB150.),together with reference to empirical formulas ( $\delta$ =8Di/1000+3) and enamel design experiences, is employed for determinations of thickness, structures and scantlings of reactor body, jacket, transmission unit, opening reinforcement, sealing and hubbed flange. As to the design of hubbed flange, finite element method, in combination with standard No.JB/T4732, titled “Stress Analysis and Design”, is used for analysis of local stress of such flange and gasket. Connecting bolts are of clamp structure, using materials of 40MnB group and 35CrMoA group, quenched and tempered. Forgings are accepted, levelII. If the pressure$\geqslant$2.5MPa,using double-headed stud Group.Gasket is of expandable graphite

compound gasket with carbon fibre around, which is pressed with expandable graphite braiding and metal corrugated ring. It has following mechanical properties: 8.5MPa to 10.0MPa for tensile strength, 20 to 30 fro Shaw hardness, 70MPa to 110Mpa for pressive strength, 41% to 50% for compression rate, 36 to 50% for resilience rate. It has small thermal expansion coefficient. Gasket factor is 1.25 and specific pressure is 5. When necessary, PTFE or other materials is applied on the gasket. Thereful, the gasket is applicable in most acid and basic cases.

Design of shaft seal is a major technical problem in the design of reactor. Double-end balanced mechanical seal is used in our design. Pressure-balancing tank is attached. The materials of motion ring and static ring vary with working medium. Silicon carbide is suitable for most working medium. Silicon carbide is high in hardness, resistant to wear, good in self-lubrication and anti-oxidation, and low in thermal expansion coefficient. Flat surface degree can be treated extremely to 0.02μm. It has mechanical properties as follows: 100HR for Shaw hardness, 60MPa for pressive strength, 500℃ for working temperature, 75 MPa·m/s for permissible pv value. It meets design requirements in actual application.

In the transmission unit,pin-cycloid planetary reducer, BLD series,is employed with electromotor capacity of 0.55kw to 15kw corresponding to volume of 10l to 10000l. Speed reducer takes double-pivot frame, HG21567-95 series.

Stirrer takes the form of gate, arm, impellor and propeller according to different media. The rotation speed usually is less than 200rpm. The straightness and vibration of shaft, and dynamic equilibirum of stirrer are specified.

It is specified in design procedures that design documents are signed by those engineers responsible respectively for design, check, standard, review and approval.

## Manufacture quality control of enamel medium-pressure reactor

1. *Choice and quality of parent materials*

Metallic materials, such as Q235-B, Q235-C, Q235-D, 20R, 16MnR and 08HT, are to be provided with quality certificates issued by steel mills. These materials are to be re-tested and also checked for appearance quality piece by piece. Q235-C and 16MnR are preferred as major materials over 16MnVR and other low-alloy or high-alloy steels from the point of view of rules, safety, enamel rigidity and economy.

2. *Preparation of porcelain glaze*

Traditional 3-in-1 ground glaze is used in our company. Finish glaze is different with process medium, which is to be resistant to acid and alkaline, and smooth in appearance. Compositions of glaze are as follows: $SiO_2$(>65%)，$B_2O_3$ (>15%)，$Na_2O$(>10%), and $Al_2O_3$, $K_2O$, CaO, MgO etc. It is resistant to acidic media except those containing HF, $F^-$, thick $H_3PO_4$ (concentration >30%，temperature>180℃), and alkaline media except strong alkaline (PH>12，temperature >100℃). The glaze resistance to alkaline and acid can be improved by adjusting concentrations of oxidants.

**Table 1** Mechanical and Chemical Properties of Acid-resistant Glaze

| Acid-resistance g/(m$^2$ • d) | Alkaline resistance g/(m$^2$ • d) | Impact energy (J) | Thermal shock (℃) |
|---|---|---|---|
| ≤0.40 | ≤3.0 | ≥230x10$^{-3}$ | ≥220 |

Enamel properties: 2.25 to 2.5 g/cm$^3$ for density, 0.40 to 0.6 X10$^5$ MPa for elastic modulus, ≤220℃

for working temperature and ≥0.87w/(m • k) for thermal conductivity.

3. *Steel plate processing*

The steel structure is designed such that the transitions are as smooth as possible and free of abrupt change. The processing of steel plate is as follows: blanking, forming (including head, nozzle, hand hole), joining, assembly, pre-heating, blasting, grinding (rough repair, fine repair).

4. *Spray coating*

Steel plate is spray-coated automatically or manually. Ground glaze is cold-sprayed and finish glaze hot-sprayed.

5. *Enamel baking*

Enamel baking is performed in vertical- or horizontal-type electrical oven with automatic temperature control. Baking temperature is between from 900 to 960℃ for ground glaze and from 880 to 940℃ for finish glaze. Spray-coating times: 5 or 6 and coating thickness each spray: 0.18mm~0.22mm. After completion of baking, enamel face is glossy and steel plate is baked completely without excessive baking. The baking process is related to temperature and time. Table 2 presents product specifications versus baking temperature and Table 3 presents product specifications versus baking time.

**Table 2** Ground Glaze Baking Temperature and Time

| Specifications | Temperature (℃) | Time (min) |
|---|---|---|
| 500L (up to 500L) | 900~925 | 21~25 |
| 1000L~2000L | 920~940 | 26~35 |
| 3000L | 930~940 | 35~38 |
| 5000L~6300L | 930~950 | 37~42 |
| 8000L | 940~960 | 43~48 |
| 10000L | 940~960 | 45~48 |

**Table 3** Finish Glaze Baking Temperature and Time

| Specifications | Temperature (℃) | Time (min) |
|---|---|---|
| Up to 500L | 880~920 | 17~24 |
| 1000L~2000L | 900~935 | 25~30 |
| 3000L | 910~935 | 26~32 |
| 5000L~6300L | 910~940 | 32~35 |
| 8000L | 920~950 | 37~40 |
| 10000L | 925~950 | 41~44 |

When baking, reactor head baking temperature and time should be a little less than that for reactor body. Baking temperature and time after 2-time glaze can be adjusted on the basis of above Tables.

6. *Metallic parts of equipments*

Shell, head, hubbed flange, manhole flange, A-and B-type nozzle and slip-on flange in accordance with HSB104-2006, with one process sheet for one work piece.

7. *Assembly*

Assembly of shell and hubbed flange, of shell and head, of upper and lower ring and inner shell, of head and hubbed flange, nozzles, manhole flange and frame base in accordance with assembly process sheet QR203.

8. *Final assembly*
In accordance with medium-pressure reactor final assembly process sheet QR203.

9. *Co-operation parts*
1) Manhole flange,hubbed flange are made of 16Mn，of 20 (Chemical elements S and P are controlled in content,S＜0.020%,P＜0.030%,especially C＜0.19%.) Forgings in accordance with JB/T4726-2002 and level II is acceptable.

2) Stirrer:
For gate stirrer, shaft straightness is below 0.10mm/m. Shaft seal, bearing and shaft neck are mounted on the shaft with concentricity below 0.05mm.The radial amplitude of vibration is below 0.15mm at mechanical seal and $0.005Ln^{-1/3}$ or below (L: length of shaft below bearing,in mm; n: rotation speed, in rpm) at shaft end in accordance with Q/320400HB003 “Technical Conditions of Enamel Pressure Vessel”.

3) Thermometer pocket tube in accordance with HSB104-2006, its straightness below 0.15%L, where L is length of pocket tube.

4) The symmetry of shaft wing is 0.15%B or below in accordance with Q/320400HB003,where B is width of shaft wing.

5) After completion of manufacture, products are to be heat-treated unless stated otherwise in the technical agreement.

6) Quality standard of clips: HSB104-2006
Material of upper clip forging: ZG45
Material of lower clip forging: 40Cr
Material of upper clip: 35CrMoA
Material of lower clip: 30CrMo
Destructive load of clip is not less than 160000N with elongation of 15% at 87.5mm as destructive. Mechanical property is quality 8.8~11 and Brinell hardness 234~285.

7) Other manufacture quality control.
Inspection sheets are prepared for manufacture processes for timely inspection according to drawings, process documents and welding procedure sheets. Besides, following standards are applicable:

a) Carbon steel and low-alloy steel plate in accordance with GB/T699, GB/T700 and GB/T6654 respectively;

b) Steel tube in accordance with GB/T8163;

c) For chemical compositions analysis of material, sampling in accordance with GB/T222 and analysis in accordance with GB/T223;

d) For tensile test, sampling in accordance with GB/T2975 and analysis in accordance with GB/T228;

e) For bend test, sampling in accordance with GB/T2975 and analysis in accordance with GB/T232;

f) NDT: Multi-echo UT is employed for testing of inner shell materials in accordance with JB/T4730.3 and level II is acceptable. Longitudinal and circumferential welds on inner shell are radiographed for 100% of weld length in accordance with JB/T4730.2 and level II is acceptable. Welds on jackets are radiographed for 20% of weld length or 250mm in accordance with JB/T4730.2, whichever is greater, and level II is acceptable table. Head nozzles made of coiled steel plate, less than 250mm in diameter, are subjected to magnetic particle test in accordance with JB/T4730.4 and level I is acceptable.

g) Testing of forgings: Clip forgings are tested in accordance with JB/T4385, level II forging, and subjected to magnetic particle test to the same extent as level II forgings hardness test in accordance with JB3965 and level III is acceptable. The testing of forgings of other parts is in accordance with JB4726.

h) For glass component and part, inspection in accordance with HG/T2637 “inspection method of geometry dimension about glass component and part”.

i) For head, test in accordance with JB/T4726;

j) For expanded graphite gasket of equipment and nozzle, inspection in accordance with HSB104-2006. Corrugations and radial grooves are not allowed on sealing face and laminations not allowed in through thickness. Gaskets are not allowed for re-use.

k) For double-end mechanical seal：inspection records, certificate of conformity and quality certificate are provided. Double-end mechanical seals are subject to hydraulic test and air-tightness test together with reactors.

l) Pressure test: The inner shell is subject to pressure test at 2.15 MPa (g) before applying enamel and at 1.6 MPa (g) after applying enamel. Hydraulic test is performed at 2.15MPa, followed by air-tightness test at 1.6MPa. Leakage, visible deformation and abnormal sound are not allowed during the test. Holding time of 30 minutes or longer is required in air-tightness test with no leakage.

m) Other quality inspections.

In our country, the production of pressure vessel is licensed. The manufacture licensing is to be possibly cancelled soon. After granted manufacture license of enamel products, the maker should be licensed for design and manufacture of corresponding class pressure vessel. Enamel medium-pressure reactor is classed as III class vessel in accordance with “Supervision Regulations of Pressure Vessel Safety and Technology” (1999 edition). Therefore licenses for A1 or A2 design and manufacture is required. So far only 3 companies are qualified for design and manufacture of enamel medium-pressure reactor in the enamel glass industry.

Total quality management is core of company management. The quality of design is the prerequisite of quality of finished product. Our company has established and improved 13 design management rules, i.e., designer qualifications, review and evaluation of designer job, job responsibility of designer, design procedures, compilation and review of design conditions, evaluation of design documents, management of design documents, revisions of design documents, re-use of design documents, specifications of design condition diagram, and use and management of design approval stamp. With reference to “Supervision and Management of Boiler and Pressure Vessel Manufacture”, our company has set up comprehensive quality management system in relation to quality control of enamel medium-pressure reactor. The system includes responsibility of manager, quality assurance

system, documents control, business contract control, design control, purchase control, process control, heat treatment control, NDT control, physiochemical test control, pressure test control, co-operative parts control, metrological equipments control, non-acceptable parts control, quality improvement, training, implementation of “Regulations of Boiler and Pressure Vessel Manufacture Licensing”.ISO19001 (ISO9001) is incorporated into our quality system.In the same time,the company standard Q/320400HB003,titled“Technical Conditions of Enameled pressure vessel”,and it’s related part standard HSB104-2006 have been established for design, manufacture and inspection. The quality control covers design, manufacture and inspection. The whole process of manufacture is monitored and traceable for the quality. The non-acceptable products are separated strictly.

In the quality system, following standards are applicable:

1. Qualification of design of pressure vessel and pressure pipeline
2. Manufacture conditions of boiler and pressure vessel
3. Pressure vessel code “regulations of pressure vessel safety technology” (1999 edition)
4. Q/320400HB003-2003—“Technical conditions of enamel pressure vessel”
5. HG2432-2001—“Technical conditions of enamel equipment”
6. GB150-98—“Steel pressure vessel”
7. GB/T222-2006—“Allowable variation of chemical compositions of steel product”
8. GB/T223-2006—“Chemical composition analysis of steel, iron and alloy”
9. GB/T228-2002——“Tensile test at ambient temperature for metallic material”
10. GB/T229-1994——“Charpy-V impact test for metal”
11. GB/T232-1999——“Bend test for metallic material”
12. GB/T699-1999——“High-grade carbon structure steel”
13. GB/T700-2006——“Carbon structure steel”
14. GB/T2975-1998——“Mechanical test specimen location and preparation for steel and its products”
15. GB/T5117-1995——“Electrode for carbon steel”
16. GB/T5118-1995——“Electrode for low-alloy steel”
17. GB/T5293-1999——“Carbon steel wire and flux for submerged-arc welding”
18. GB20613-2000——“Stud bolt”
19. GB20613-2000——“Hexagon nut”
20. GB/T14957-1994——“Steel wire for fusion welding”
21. JB/T4708-2000——“Welding procedure qualifications for steel pressure vessel”
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23. JB/T4725-1992——“Lobe-type base”
24. JB4726-2000—“Carbon steel and structure steel forgings for pressure vessel”
25. JB/T4730-2005—“NDT for pressure-containing equipment”
26. JB/T4732-2005——“Stress analysis and design”
27. JB4744-2000—— “Mechanical test for production test plate of steel pressure vessel”
28. JB/T4746-2002——“Standard head”
29. HSB104-2006——“Standards for manufacturer’s parts”
30. Other standards

In present, enamel medium-pressure reactor is widely used in medicine industry, chemical industry, light industry, food processing, dyeing and printing, and scientific research etc. As we know, enamel reactors have been used in Nanjing Huaan Drug Company,Liaoning Huaduan Drug Company, Jiangsu Dengguan Pesticide Company, Zhenjiang Agrochemical Company, Zhejiang Xinganjiang Sanmu and Jingtan Biochemical Institute etc. More and more enamel reactors are expected in use as the economy is growing and new materials are emerging.

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