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Report:

From noise to information: a new technology of olefin polymerization fluidized bed reactor based on acoustic emission*

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Abstract: The fluidized bed is widely used in many industrial processes because of its vigorous mixing and heat transfer properties. However, when heat transfer is blocked, the particles are easily melted and agglomerated, and even cause the industrial reactor to shut down. From the point of mechanism analysis, the process of explosive agglomeration is a typical meso-scale problem in the fluidized bed, and there is a complex evolution process between particle fluidization and reactor shutdown. Grasping the regulation of meso-scale structure is one of the major challenges faced by chemical engineering. Thus, in this background, the fluidized bed acoustic emission detection technology, agglomeration fault self-repair technology, and a direct scale-up technique of the fluidized bed mathematical model were invented. These technologies have provided strong reliability for stable operation and have been successfully applied in 14 sets of industrial plants.

Key words: Fluidized bed; Acoustic emission; Agglomeration; Meso-scale; Scale-up
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1 Research background and technical challenges

Of all the global synthetic resins, polyolefin production has topped the list, and played an important role in national economic construction and social development. In China, polyolefin plant capacity was more than 25 million tons in 2014, whereby the replacement of paper products is equivalent to saving half of the forest area of Zhejiang Province annually. Moreover, the polyolefin product manufactured as agricultural film is an effective expansion of China's effective arable land area, as much as 6.67×10^4 km^2. Polyolefin is an important foundation of the national economic construction materials and as it contains only carbon and hydrogen elements, it is easy to recycle and benefits environmental protection. Polyolefin is a strong factor in easing the tension between China's population and maintaining an ecological balance. However, at present the self-sufficiency rate of China's polyolefin is only 70%. China's polyolefin production capacity ranks second in the world, and 40% of this is completed by the fluidized bed polymerization reactor.

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When heat transfer is blocked, particles are easily melted and agglomerated, and may even cause the industrial reactor to shut down. The economic loss, in these cases, is particularly large, and mainly is a consequence of bursting agglomeration. This has become a major technical problem which has plagued the stable operation and the scale-up of the reactor for a long time. At present, ExxonMobil, Dow, Lyondellbasell, Borstar, etc., are the major international producers of polyolefins, through reorganization, large-scale and high-efficiency production to reduce production costs and improve competitiveness. In mainland China, polyolefin production process technology has been dependent on foreign imports. Not only that, the lack of domestic technologies to improve the operational reliability of the production plant, scale-up design, process monitoring, and operation optimization technology result in a long-term profitability lower than foreign competitors. With this as background, this project invented the technology of the polyolefin fluidized bed reactor based on acoustic emission monitoring, and has successfully applied it in the 300 000 t/a ethylene polymerization fluidized bed reactor in Tianjin, China, which is part of the state "Eleventh Five-Year" key construction project. The design and construction has expanded to 14 sets of large-scale olefin polymerization plants, and achieved the efficient production of polyolefins. This is also recognized as an important symbol for the synthetic resin industry in China and has been listed as the national science and technology support project many times.

In solving the problem of agglomeration in the process of polymerization, there are three difficulties: (1) the *in-situ* characterization of particles and agglomerates in the fluidized bed is changeable and they are difficult to detect; (2) the on-line intervening mechanism of particle melting agglomeration is unclear and difficult to control; (3) strong coupling of flow and heat transfer process and the reactor scale-up law is unknown, making it difficult to scale up the reactor. Through years of laboratory experiments and industrial applications, we find that the key to solve the three difficulties lies in a detection technology breakthrough. Fortunately, we found that acoustic emission signals generated by the collision of fluidized particles and the reactor wall surface can be possible to detect and reflect the changes in particle characteristics. We try to solve these three difficulties and the general idea of the invention is listed as follows:

In response to the first difficulty, we developed fluidized bed acoustic emission detection technology.

For the second difficulty, we invented the fluidized bed agglomeration fault self-repair technology.

Aiming at the third difficulty, a mathematical model of direct scale-up technique of the fluidized bed was invented.

The first invention, via acoustic emission, provides a new platform technology for fluidized bed design and research. The second invention enhances the stability of the plant from the operational level, and the third invention provides reliability at the design level. With the support of a series of national key projects, Zhejiang University and SINOPEC Qilu Petrochemical Company of China combine the production and research in close cooperation. After 22 years of unremitting efforts to overcome the three major problems, the access to a series of innovative inventions is achieved.

2 Technical innovation and details of the inventions

2.1 Acoustic emission detection technology

In the complex flow of the fluidized bed environment, given the lack of *in-situ* detection technology and effective information extraction, we completed the following two tasks.

In the first task, a multi-scale analysis method of acoustic emission signals was established. The acoustic emission signals were decomposed into micro-scale, meso-scale, and macro-scale signals by wavelet decomposition, rescaled range analysis (R/S) classification in terms of Hurst's rescaled range, and reconstruction. Thus, the corresponding relationship between multi-scale of acoustic signals and multi-scale of flow structure was established, which solves the problem of effective information extraction.

Zhao and Yang (2003) applied the concept of fractional Brownian motion to analyze the pressure fluctuations in a gas solid fluidized bed, namely in terms of Hurst's rescaled range (R/S analysis) as a criterion of the multiscale resolution of pressure fluctuations. The Hurst analysis was recognized as a tool for flow-regime identification and classification, in which the Hurst exponent H was used as a descriptor to the scaling behavior of fractal curves, called self-similarity (Maucci *et al*., 1999). After dividing a time series of length N into m subseries with length τ, for each subseries, (1) compute standard deviation S_k, (2) normalize the data $x_{i,k}$ with sampling data mean $x_{i,k}-E_k$ for i=1, 2, …, τ, where E_k is the mean for each subseries, (3) the cumulative deviation $\sum_{j=1}^{i}(x_{j,k}-E_k)$ for i=1, 2, …, τ is obtained, (4) look up the range for the difference between maximum and minimum of $\left\{\sum_{j=1}^{1}(x_{j,k}-E_k), \sum_{j=1}^{2}(x_{2,k}-E_k), \cdots, \sum_{j=1}^{\tau}(x_{j,k}-E_k)\right\}$, namely R_k, and (5) finally rescale the range R_k/S_k.

Coupled with R/S analysis, a wavelet transform is developed for time frequency analysis to deal with signal analysis and its processing (Zhao and Yang, 2003; He *et al*., 2009). Compared with the traditional Fourier method, there are some important differences. Wavelet analysis is applied to transient signal processing, whereas the Fourier transform fails to reveal the local turbulence. We found that pressure fluctuations caused by different factors can be resolved from measured signals by means of Hurst analysis. For example, micro-scale interaction pressure signals representing the dynamics of individual particles can be obtained by synthesizing the detail signals in levels 1 and 2 because of their similar fractal feature. Pressure signals of meso-scale interaction representing the dynamics of the dense phase and the bubbling phase can be obtained by synthesizing the detail signals in levels 3–9 because of their similar bifractal feature. Level-9 approximation signals, representing the global system of particle-fluid suspension within its boundaries, are macro-scale signals. An example of industrial signal rescale is summarized in Fig. 1 (Zhao and Yang, 2003), where d0 is the original signal, d1–d7 are the detail functions, and a1–a7 are the approximation functions for each level. The theory is not described here, and for details, please refer to He Y.J. *et al*. (2009) and He L.L. *et al*. (2016).

In the second task, a new technique for the acoustic emission measurement of particle parameters was developed, which included 14 parameters including micro-scale particle size distribution, mesoscopic granular agglomeration, and macroscopic particle flow pattern. This has realized real-time on-line measurement of the particle parameters of the fluidized bed reactor (Yang *et al*., 2003).

In particular, acoustic emission detection of the fluidized particle size distribution was achieved on-line every 10 s, much faster than the traditional sampling method of 2 h. The early warning method of acoustic emission was also established. Compared with the traditional temperature and pressure warning methods, the early fault warning time of this technology was 2.5 h ahead of time and at least half an hour ahead of the γ-ray detection technology.

It is found that the evolution of the granular flow in the reactor can be measured and analyzed by the acoustic emission signals generated by the friction and collision between the particles and the walls of

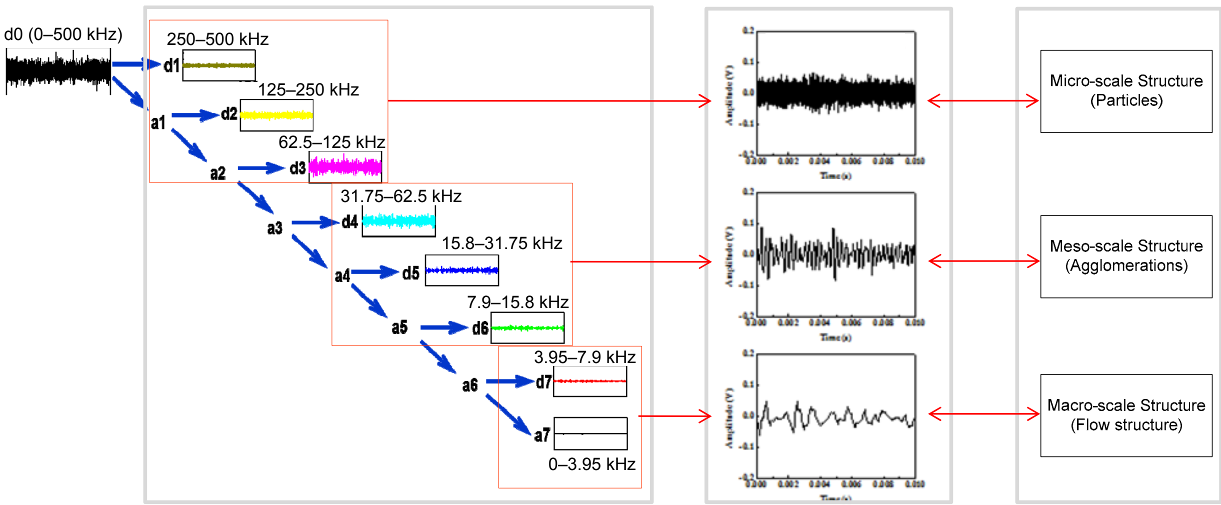

Fig. 1 An example of acoustic emission signal rescale by applying wavelet and R/S analyses (Zhao and Yang, 2003)

the reactor. This is the first time it has been possible to correlate and calibrate the acoustic emission signals in different frequency bands with the particle flow parameters (Yang *et al.*, 2003) in this project, as shown in Fig. 2 (Zhao and Yang, 2003; He *et al.*, 2009). The on-line detection technology of particle size and particle size distribution (PSD) was established on the basis of the experimental data (Wang *et al.*, 2008) and other parameters. In detail, unique on-line detection of particle agglomeration (Zhou *et al.*, 2011), particle fluctuation velocity (Wang *et al.*, 2010), and dew point temperature was developed. Through the spatial distribution of energy parameters, as detected in Fig. 3 (Wang *et al.*, 2009b), produced by acoustic emission of the wall, circulation flow pattern related with particle size, wall sheeting of fine powder, bias current flow, high electrostatic region positioning method and the resulting industrial fluidized bed electrostatic distribution control method are also established (Yang *et al*, 2009). Moreover, through acoustic emission monitoring, additional parameters of more than 10 species are studied and developed.

Industrial applications show that the acoustic emission measurement method has high precision in measuring solid particle motions (Jiang *et al.*, 2007) and particle size distribution in fluidized bed in Fig. 4 (Yang *et al.*, 2005; Wang *et al.*, 2010), agglomeration with measurement error less than 10% (Yang *et al.*, 2003; Wang *et al.*, 2009a), where d_{p1}–d_{p7} are particles with different sizes and ω_1–ω_7 are the volume fractions for each particle. Acoustic emission technology not only changed the traditional artificial sampling analysis, but also greatly reduced the blind area of the

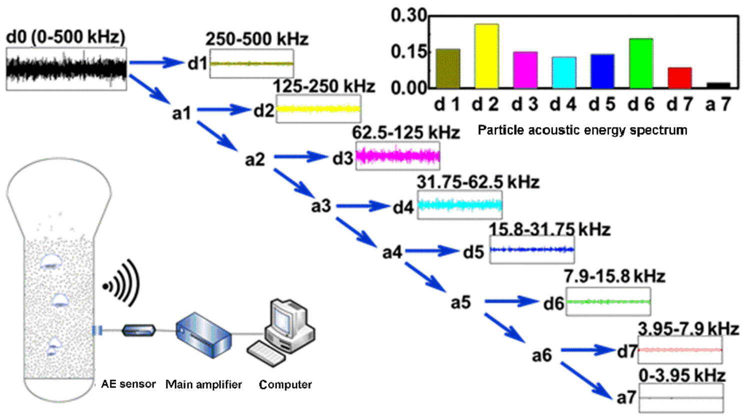

Fig. 2 Acoustic emission measurement system and signal multi-scale resolve analysis method (each particle size with a characteristic energy spectrum; AE: acoustic emission; summarized from Zhao and Yang (2003) and He *et al.* (2009))

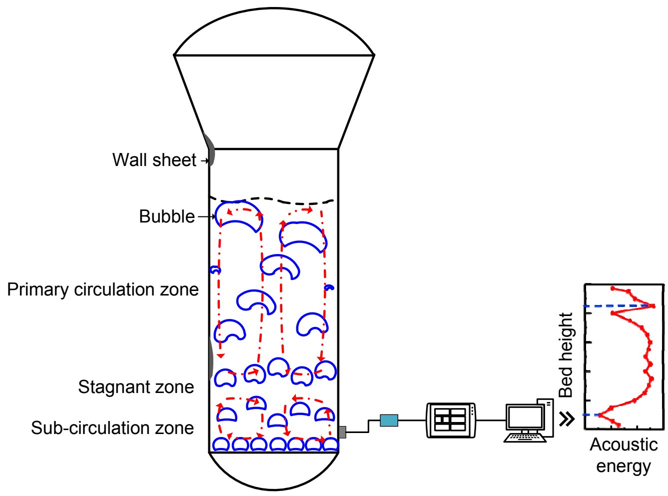

Fig. 3 Corresponding relationship between the two-cycle particle circulation flow pattern and the acoustic energy axial distribution (Wang *et al.*, 2009b; 2010)

gas phase polymerization process. This reactor operation monitoring technology has the character of innovative, advanced functions, and applicability for industrial application.

As stated above, the invention is based on the measurement and analysis of the acoustic emission information, and establishes a new means for efficiently monitoring particle motions, which is different from the traditional method of designing and scaling up the reactor based on the temperature and pressure variables (Wang *et al.*, 2007; Ren *et al.*, 2008). This satisfies new requirements for process development and operational optimization of fluidized bed reactors considering its main function of solid processing. Not only that, through the on-line and non-invasive acoustic emission technology, the detection of particle motion information is accomplished in industrial plant. Thus, the corresponding relationship between a theoretical model and industrial devices is achieved (Wang *et al.*, 2010), which accelerates the continuous improvement and optimization of industrial technology (Yang *et al.*, 2008; Wu *et al.*, 2013).

By acoustic emission and electrostatic detection, all of the charge separation, the corresponding electrostatic wall surface enrichment effect, and the electrostatic distribution with the positive charge upwards and negative charge downwards in the polyolefin fluidized bed were found (Fig. 5) (Wang *et al.*, 2008). In Fig. 5, the equipotential line is the area with the same electrostatic potential. What is more, the double circulating flow pattern of solid particles has been recognized and a new platform has been developed for the design and optimization.

2.2 Reactor operation optimization technique

The present methods for the operational optimization of the polyolefin fluidized bed reactor do not provide for appropriate on-line intervention in agglomeration. Moreover, the risk of on-line repair after agglomeration is high, and precise quantitative monitoring technology is lacking (Ren *et al.*, 2011). Thus, we propose a reactor operation optimization technique based on acoustic emission detection, which has two aspects:

A soft measurement method of condensate flowrate based on acoustic emission detection was developed. We used acoustic emission detection to get the dew point temperature of the circulating gas, so as to do component correction, and to achieve the condensate flowrate and accurately measure other key parameters. The process parameters, thermodynamics, polymerization kinetics, and computational fluid dynamics (CFD) simulation were used to establish the monitoring system for the production process, and this effectively solved the problem of the lack of accurate quantitative monitoring technology. The flow diagram is shown in Fig. 6.

In the industrial process, the polymer powders can block the distribution plate and the heat exchanger tube leading to device shutdown for maintenance. This has been the key technical problems. Combined with acoustic emission agglomeration

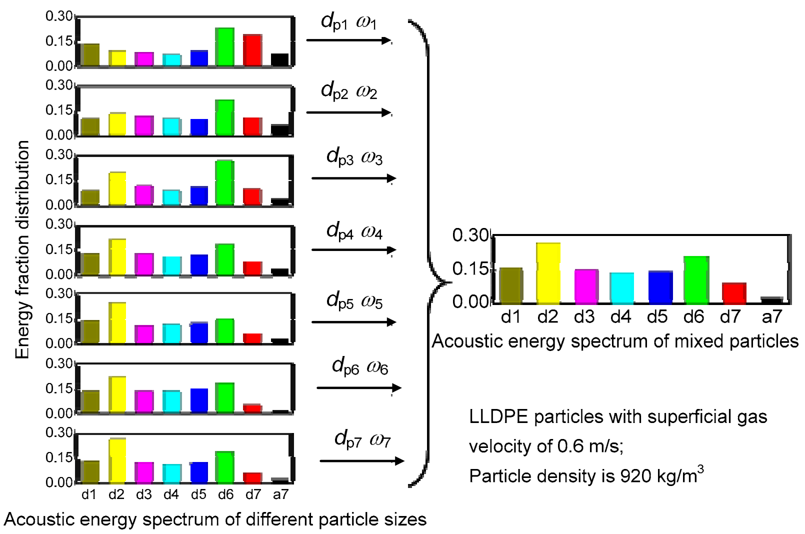

Fig. 4 On-line particle size distribution detection technology (LLDPE: linear low density polyethylene) (Yang *et al.*, 2005)

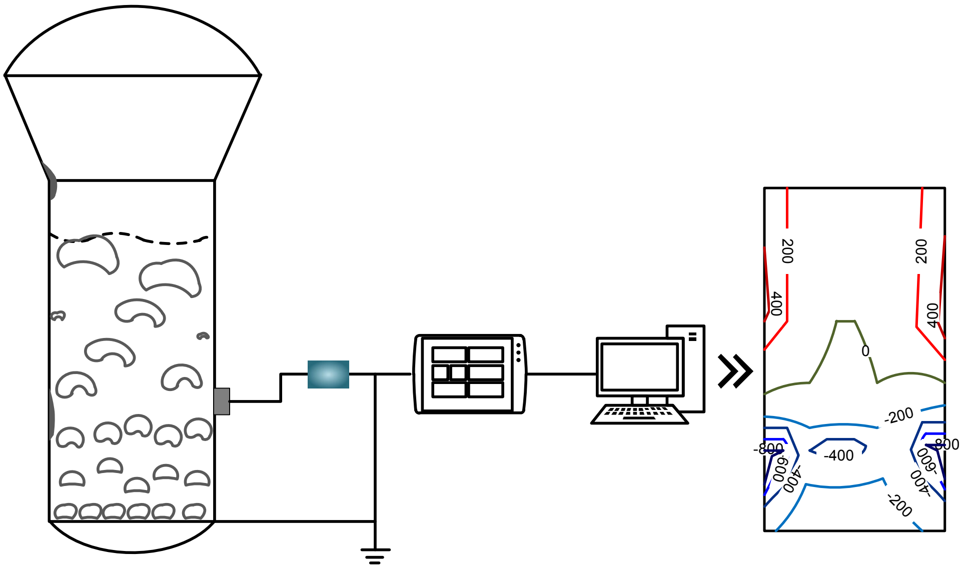

Fig. 5 Electrostatic potential distribution in polyethylene fluidized bed (unit: V) (Wang *et al.*, 2008)

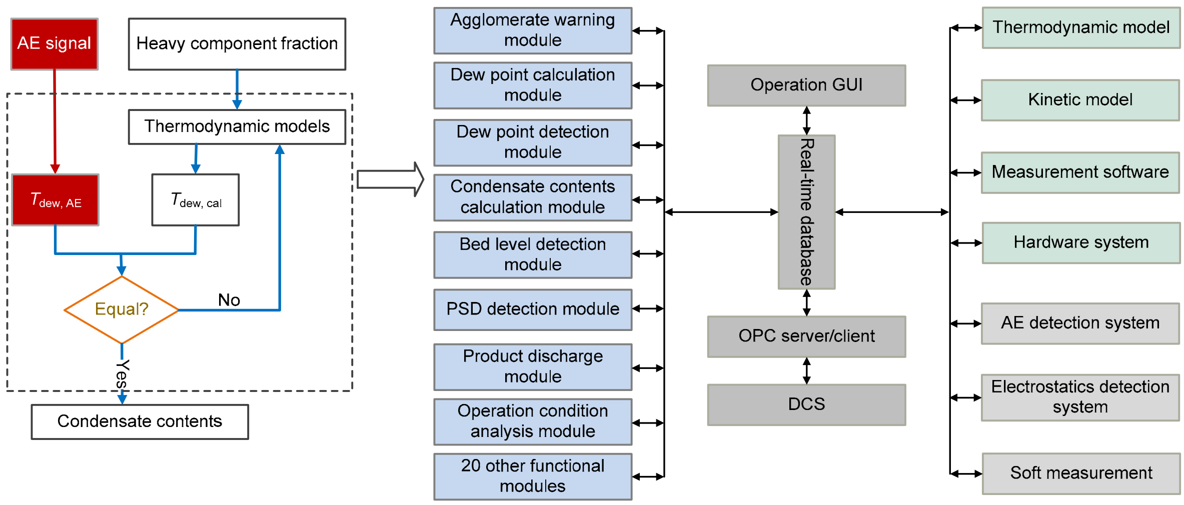

Fig. 6 Monitoring system for the production process of the polymerization fluidized bed reactor combining acoustic emission detection, thermodynamics, polymerization kinetics, and CFD simulation

$T_{dew,AE}$: dew point temperature detected by acoustic emission; $T_{dew,cal}$: calculated dew point temperature; GUI: graphical user interface; OPC: object linking and embedding for process control; DCS: distributed control system

early warning and production process monitoring, our group invented a condensate on-line self-repair system. With the help of the monitoring system, once the distribution plate blockage and other faults give an alarm, a specified amount of condensed liquid will be introduced, and accurate control of the flushing flowrate can be effected. By this means, the distribution plate will be washed out, agglomerate will fall off and be discharged from the reactor and thus the reactor will not so easily shut down and on-line washing and self-repair will have been achieved.

The invention is based on the method of detecting the volume of condensate and the gas drift by acoustic emission. The condensate of the process used to clean the distribution plate and operation of the heat exchanger by the circulating gas are also developed for the first time (Han *et al.*, 2015). Industrial applications show that the polymerization unit operates smoothly and can be automatically cleaned during the production cycle as illustrated in Fig. 7. The operating cycle can be extended to 38 months, an increase of nearly 50%.

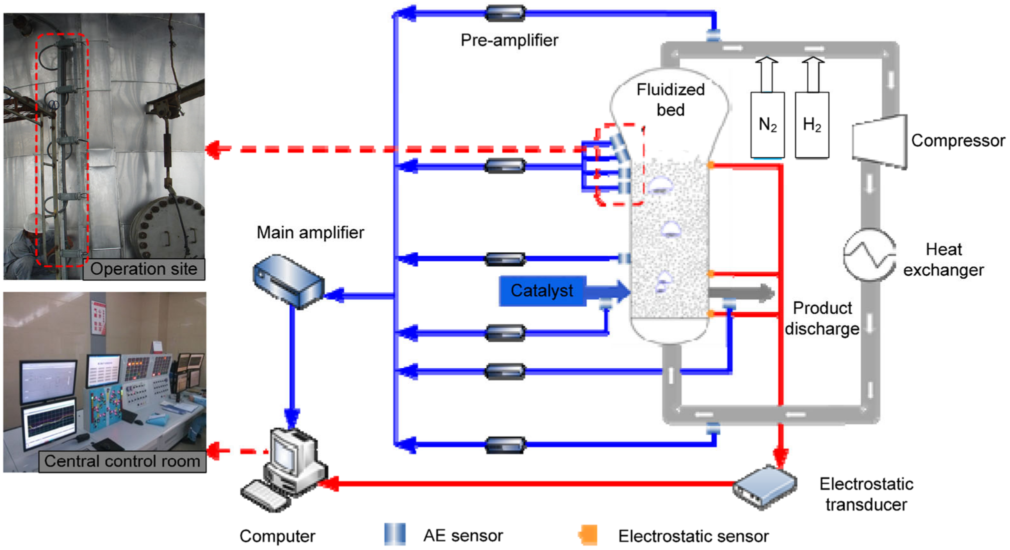

Fig. 7 Gas phase polyethylene process monitoring system with acoustic emission and electrostatic detection

Different from the conventional technique of monitoring the condensed operation based on an equilibrium thermodynamic model, the present invention calculates the condensate compositions and the condensate fraction in a non-equilibrium state using a modified method of acoustic emission dew point correction, creating a new monitoring process. This improves the monitoring accuracy of the operating parameters of the condensing process. It is applied in six sets of large-scale polyethylene fluidized bed units under the SINOPEC (Yang *et al.*, 2006; Wang *et al.*, 2008a; Wang *et al.*, 2011). The application shows that the operation level of the equipment is improved and the comprehensive performance index is at a world leading level.

2.3 Reactor scale-up technique

This invention includes a new technology of structural scale-up of the polyolefin fluidized bed reactor. It aims to tackle the problem of strong coupling of flow-heat transfer process and unclear scale-up of the reactor. We propose a reactor scale-up technique based on acoustic emission detection, which has two aspects:

A direct scale-up method based on acoustic emission detection was developed. We use the acoustic emission technique to obtain the important data of the critical space-time yield of different sizes. The mathematical model of the space-time yield (STY) is established with the scale-up law of equal STY and operation law of unequal STY, achieving a direct scale-up of the reactor. As shown in Fig. 8, under the condensing model, STY increases significantly, and the designed STY and heat transfer coefficient can be calculated by the following equation:

$$\frac{\mathrm{STY}}{T_{\mathrm{e}}-T_{\mathrm{f}}}=\eta[(1-\mathrm{e}^{-KH})(u-u_{\mathrm{mf}})+u_{\mathrm{mf}}], \tag{1}$$

where T_{e} and T_{f} are the emulsion phase temperature and the reactor inlet temperature, respectively, K is the heat transfer coefficient, η is the heat transfer efficiency, and u and u_{mf} are the superficial gas velocity and minimum fluidization velocity, respectively. Eq. (1) is obtained based on the heat balance in the fluidized bed using a two-phase model. By integrating the polymerization heat, heat transfer between the emulsion and bubble phases, heat loss of gas in emulsion and solid discharge, and condensate vaporization heat, the relationship between STY and the operation parameters can be estimated.

$$K=\frac{H_{\mathrm{be}}^{*}}{u_{\mathrm{b}}\rho_{\mathrm{g}}C_{\mathrm{pg}}}, \tag{2}$$

$$\eta=\frac{\rho_{\mathrm{g}}C_{\mathrm{pg}}}{(\Delta H_{\mathrm{r}}-Q')H_{\mathrm{bed}}}, \tag{3}$$

where H_{be}^{*} is the critical heat transfer coefficient, u_{b} is the bubble velocity, ρ_{g} is the gas density, C_{pg} is the gas specific heat capacity, H_{bed} is the bed height, ΔH_{r}

is the polymerization heat, and Q' is the heat by adding solid discharge heat loss, condensate vaporization heat, and heat loss at the wall.

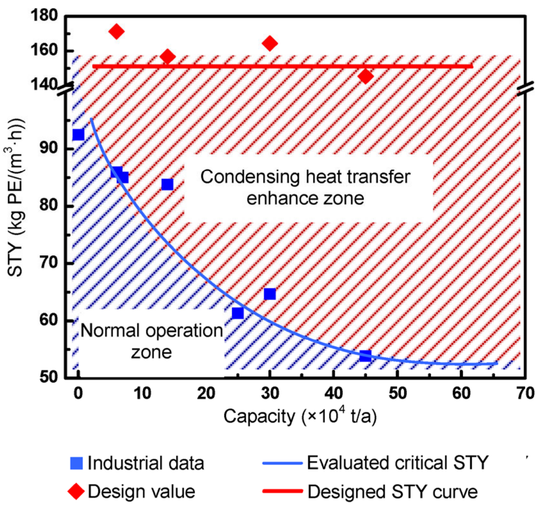

Fig. 8 Different operation zones under normal conditions and condensing mode

The uniform distribution of the flow field technique in the reactor based on acoustic emission detection was invented. On the one hand, we use the acoustic emission technique to detect the particle drift flow, design a multiple distributor with anti-particle deposition, and reliably achieve the uniform distribution of gas-liquid-solid three phases. Thus, the flow dead zone of the reactor is eliminated. On the other hand, we also use the acoustic emission technique to detect the particle double circulation flow pattern, design the best installation position of the high active catalyst injectors, and realize the efficient dispersion and homogeneous mixing of the catalyst.

A new gas-liquid multi-homogenization technique for large-scale polymerization fluidized bed reactor was invented and designed by the combination of CFD simulation and experiment (Fig. 9), including the necking structure on the bottom inlet pipe (Wang *et al.*, 2012), a distribution plate (Yang *et al.*, 2004) that realized the planar purging function by pressure drop redistribution, and acoustic emission sensor for the distribution plate, achieving a real-time monitoring system of agglomeration and the liquid accumulation condition (Wang *et al.*, 2008b). For instance, the traditional fluid guiding director is a straight stand structure, which results in the direct fluid flow into the reactor and nonuniform fluid distribution. By improving the structure into gradient slides, the uniform fluid distribution is guaranteed. Moreover, the distribution plate is also improved into a triangular form from the traditional opening hole structure, which ensures that the fluid can effectively clean the sediment particles and prevent agglomerate from forming at the plate. The industrial application shows that the new technology can fully guarantee the uniform dispersion of gas and liquid, effectively prevent the occurrence of biased flow in the reactor, and greatly enhance the ability of the distribution board. As a result of guaranteeing the heat transfer and flow similarity, the risk of explosive agglomeration in polymerization reactor was substantially decreased. Compared with the equipment adopting imported technology, ethylene monomer consumption was reduced by more than 0.84% and energy consumption fell more than 24%. For the industrial plant of the 300 000 t/a ethylene polymerization fluidized bed reactor in Tianjin, the saved monomer can be as much as about 2520 t/a, which is recognized as a significant technical improvement for the industrial applications.

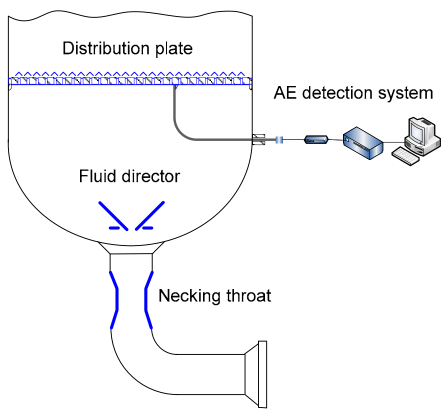

Fig. 9 Multi-homogenization technique for large-scale polymerization fluidized bed reactor (Yang *et al.*, 2004; Wang *et al.*, 2012)

3 Industrial applications and achievements

The newly invented technology has been successfully used in the 300 000 t/a ethylene polymerization fluidized bed reactor in Tianjin as the state key project of "Eleventh Five-Year".

This indicates that China's gas phase polyethylene industry, for the first time, has a complete set of domestic technology. The results show that the main technical and economic indices, such as total monomer consumption, catalyst consumption, and comprehensive energy consumption, have reached an internationally advanced level, implying the full satisfaction of the long-term operational requirement and a reliable auxiliary equipment for the downstream ethylene process. The operating cycle can be extended to 38 months, an increase of nearly 100%, given that the traditional process can smoothly operate for about 18 to 20 months (Fig. 10).

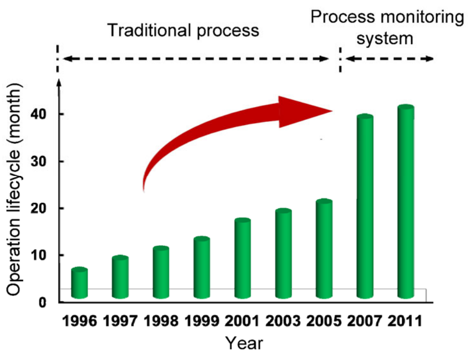

Fig. 10 Operation lifecycle of gas phase ethylene polymerization reactor (data from Tianjin Petrochemical Engineering Co. Ltd, SINOPEC)

The design and construction has expanded to 14 sets of large-scale olefin polymerization plant, and achieved the efficient production of polyolefins. This is also recognized as an important symbol for the synthetic resin industry in China and has been listed as the national science and technology support project many times.

The invention has been applied to the design or modification of 14 sets of large-scale polyolefin plants, and the polyethylene production capacity is up to 2.63 million tons, accounting for more than 50% of the production capacity of polyethylene in mainland China. As stated before, compared with the equipment adopting imported technology, ethylene monomer consumption was reduced by more than 1.57% (from 1.020 to 1.004 t/t) and energy consumption fell more than 25.2% (from 138.86 to 103.08 kgEO/t, data from the National Polyolefin Resin Industry Organization Secretariat, China).

The present invention solved the problem of bursting agglomeration and the scale-up of the reactor, so that the reliability of the device is remarkably improved. This new technology has been widely used in polyethylene, polypropylene fluidized bed production, resulting in considerable economic and social benefits.

The reactor design and operational optimization technology, from the point of view of methods and design principles, are fully applicable to a variety of gas-phase polyethylene and polypropylene technology. However, the extension to slurry polymerization and solution polymerization process is still under way, and research is still required on the metallocene catalyst reactor.

The polymerization reactor design and optimization technology established in this project aims to solve the problem of long-period operation and reliability, and is appropriate for large-scale engineering construction and scaling up.

Future work will look at polymer structure design and regulation, especially the research and development of new detection methods that are parameter-sensitive in the industrial application.

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Introducing editorial board member:

Prof. Jing-dai WANG joined Zhejiang University, China in 2002 as an assistant professor of College of Chemical and Biological Engineering when he finished his PhD. During 2002 to 2004, he worked on the confined polymerization of olefine and micro-channel-extruder mixing in marcomelecular system and fluidization of the gas phase fluidized bed. Meanwhile, his interest also extended to the preparation of bimodal polyethylene by oscillating operation of hydrogen. He worked as an associate professor during 2005 to 2009, when his research mainly focused on the multi-scale structuration and experimental characterization for multi-phase reactive system, design of micro-reactor architecture for olefine polymerization and control of polymer structure, high activity catalyst for ethylene polymerization, and sheet orientation and measurement in the gas phase fluidized bed polymerization reactor by acoustic emission technique.

Until now, Prof. WANG has won the New Century 151 Talent Project (the first echelon) of Zhejiang Province and is selected by the New Century Excellent Talents Supporting Project of Ministry of Education, China. He has received the National Science Fund for Distinguished Young Scholars of China.

中文概要

题　目： 从噪声到信息：基于声发射监控的聚烯烃流化床反应器新技术

目　的： 在全球所有的合成树脂中，聚烯烃的产能位居榜首，在国民经济建设和社会发展中发挥着重要作用。但流化床聚合反应器内的高静电会引起颗粒团聚，一旦传热受阻，颗粒极易熔融结块，甚至导致装置爆聚停车。本研究旨在针对流化床聚合反应过程具有强放热、高静电、变粒径的特点，提出基于声发射技术的多种检测手段和信息提取方法。

创新点： 针对聚合过程的爆聚结块问题，本研究主要解决了以下难题：（1）流化床内颗粒和结块的原位表征技术缺失，难以检测；（2）颗粒熔融结块过程的在线干预机制不清楚，难以调控；（3）流动传热过程强耦合，反应器放大规律不明，导致反应器难放大等问题。

方　法： 创立声发射信号的多尺度解析方法，通过小波分解和重标极差分析法分类重构将声发射信号分解为微尺度、介尺度和宏尺度信号；提出基于声发射检测的反应器操作优化技术，并通过建模确立流化床聚合反应器的时空产率数学模型。

成　果： 新技术成功应用于“十一五”国家重点建设项目——天津百万吨乙烯工程相配套的年产 30 万吨气相法聚乙烯流化床反应器的设计建设。并且，新技术已成功应用于 14 套大型聚烯烃装置的设计或改造，所涉及的聚乙烯产能达 263 万吨。作者所在团队也因该技术发明获得了 2016 年度国家技术发明奖二等奖。

关键词： 流化床；声发射；聚团；介尺度；放大