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水煤浆热解过程中 HCN 和 NH_3 释放特性的分析

孟德润, 赵　翔, 周俊虎, 岑可法
(浙江大学 能源洁净利用国家重点实验室, 浙江 杭州 310027)

摘　要: 采用固定床反应器对水煤浆及其制浆原煤在惰性气氛下, 制浆原煤在水蒸气气氛进行热解试验, 研究 HCN、NH_3 的释放特性。结果发现, 制浆原煤和水煤浆 HCN 的释放量, 随温度增加变化缓慢, 趋于稳定; 而制浆原煤在水蒸气气氛下, HCN 的量随温度变化增加迅速, 析出量远远超出制浆原煤及水煤浆的释放量。制浆原煤 NH_3 释放量随温度升高先增加后有下降, 在 1 000 ℃左右出现一个峰值, 温度继续升高, NH_3 的量不再增加反而开始降低; 水煤浆中 NH_3 析出的量随温度增加, 虽变化缓慢, 但是仍比制浆原煤释放出的 NH_3 量要多; 制浆原煤在水蒸气气氛下, 随温度的升高, NH_3 的量一直呈增加趋势, 温度到达 1 000 ℃后, 增加更加迅速, 释放量大于制浆煤和水煤浆的 NH_3 释放量。综合考虑 HCN 和 NH_3 的释放量以及燃料的着火、燃烧, 显然水煤浆燃烧要优于煤粉的单独燃烧和煤粉喷水蒸气燃烧。

关　键　词: HCN; NH_3; 热解; 水煤浆

中图分类号: TQ534.4　　文献标识码: A

1　前　言

近年来, 各国的研究者为了弄清燃料型 NO_x 的生成和破坏进行了大量的理论和试验研究工作, 美国、日本、澳大利亚、芬兰等国家都是较早研究煤热解条件下氮的化学行为的国家[3]。我国对煤中氮行为的研究主要集中在降低 NO_x 上。

水煤浆作为一种新型燃料在我国已完成了试验室和工业性试验阶段, 目前已在大中型电站锅炉上成功应用, 并取得了比较好的经济效益和社会效益。但对水煤浆中 NO_x 排放缺乏基础性研究。本文主要采用固定床反应器对水煤浆及其制浆原煤在惰性气氛条件下, 制浆原煤在水蒸气气氛进行高温热解试验, 研究热解温度、水分对煤中燃料氮析出特性的影响, 特别是对 NO_x 两种重要前驱物 NCH、NH_3 析出特性的影响。

2　试验内容及装置

在惰性气氛条件下, 采用固定床反应器对水煤浆及其制浆原煤、制浆原煤水蒸气气氛(由 Ar 气携带水蒸气)进行 800 ℃、1 000 ℃、1 200 ℃热解试验, 研究热解温度、水分对样品中 HCN、NH_3 析出特性的影响, 实验样品的元素分析、工业分析及灰成份见表 1 和表 2。试验装置如图 1 所示。

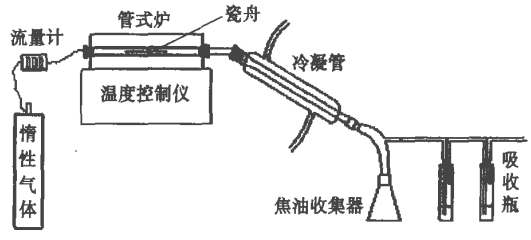

图 1　试验系统装置示意图

样品(0.5 g)均匀分布于长 97 mm 的磁舟里, 送入高温定碳炉陶瓷燃烧管(φ23 mm×600 mm)的高温段(90 mm)中进行加热。石英管内通有 99.99% 的氩气, 氩气量为 100 mL/min。在规定的热解条件下加热, 加热速率为 100 ℃/min, 热解温度由 KSY 智能型温度控制仪控制。热解产生的气态产物先通过冷凝管进入焦油收集器, 再分别进入两只装有 50 mL 浓度为 0.2 mol/L NaOH 和两只 50 mL 浓度为 0.005 mol/L H_2SO_4 吸收液的串连多孔吸收瓶中, 进行 HCN、NH_3 的吸收。为保证 HCN、NH_3 互不干扰和试验的精确度, 采用双管同时加热并行吸收的方法进行试验。最后取出磁舟, 收集煤焦, 对收集到的焦油、HCN、NH_3 及焦炭分别进行分析。

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作者简介: 孟德润(1979－), 男, 河北沧州人, 浙江大学博士研究生.

对于试验中收集到的焦油、HCN、NH_3 及焦炭, 采用了不同的方法进行分析。其中, 焦油中的氮通过化学法测定(GB/T 476－91), 焦炭中的氮通过 CHNS－932 元素分析仪测定。而对于 NO_x 生成的重要前驱物 NH_3、HCN, 分别采用了水杨酸－次氯酸盐分光光度计法和硝酸银滴定法进行分析。

表 1　试验样品的元素分析与工业分析

	元素分析/%						工业分析/%			
	C_{daf}	H_{daf}	N_{daf}	S_{daf}	O_{daf}	N/C	M_{ar}	A_{ar}	V_{ar}	F_{car}
原煤	85.63	5.65	1.49	1.77	5.46	0.017 39	6.83	5.95	32.48	54.73
煤浆	84.36	5.22	1.77	1.27	7.38	0.020 94	34.14	7.75	23.81	34.30

表2　试验样品的灰成份分析

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2
原煤	46.94	38.03	7.84	3.76	1.07	0.59	0.78	1.01
煤浆	38.91	44.80	7.23	3.18	1.16	2.64	0.79	1.30

3　实验结果及分析

3.1　HCN 随温度变化的规律

图2 为样品热解析出 HCN 随温度变化释放规律图, 制浆煤加水表示制浆煤在水蒸气气氛下热解的曲线(下同)。从图中可以看出, 制浆原煤和水煤浆 HCN 的释放量均增加, 随温度增加变化缓慢, 趋于稳定, 但水煤浆中 HCN 的释放量要比制浆原煤高 15%左右。而制浆原煤在水蒸气气氛下, HCN 的量随温度变化增加迅速, 而且 HCN 的析出量远远超出水煤浆。

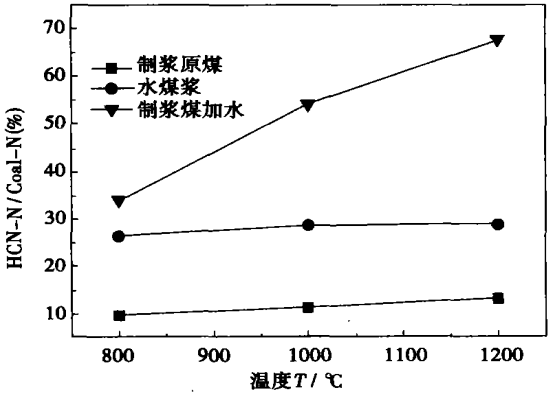

图 2　温度对HCN 释放的影响

水煤浆热解析出 HCN 比制浆原煤多, 原因可能有以下几个: 一是其含氮量比制浆原煤高一些; 二是水煤浆热解时, 含有大量的水分首先汽化为水蒸气, 水蒸气会打开封闭的孔隙, 焦的表面积和孔容积变大, 温度越高, 扩孔作用越明显, 这有助于减小挥发分氮析出的阻力, 有利于挥发分的释放, 使其挥发分含量高, 而挥发分是 HCN 的主要来源; 三是水煤浆热解试验中基本没有收集到焦油, 可能是焦油进一步裂解生成了 HCN。因此水煤浆的 HCN 含量高于制浆原煤。

而制浆原煤在水蒸气气氛下, HCN 释放量比水煤浆还要大, 其原因可能是水蒸气气氛下可以提供的 H 自由基要远远多于水煤浆中水分汽化产生的 H 自由基, 另外水煤浆中的大量钠离子对 HCN 的形成也有一定抑制作用。

3.2　NH_3 随温度变化的规律

图 3 为样品热解析出 NH_3 随温度变化释放规律图。制浆原煤 NH_3 的释放量随温度升高先增加后有下降, 在 1 000 ℃左右出现一个析出峰值, 原因可能为 NH_3 主要来源于季氮[3], 而季氮在 1 000 ℃左右就分解比较完全, 另外 NH_3 在高温下又可以发生二次反应, 因而当温度达到 1 000 ℃左右后, 温度继续升高, NH_3 的量不再增加反而开始降低, 同时由于煤中季氮的含量较低, 而吡咯型氮、吡啶型氮比例高, 也使得热解产物中 HCN 的含量比 NH_3 高。

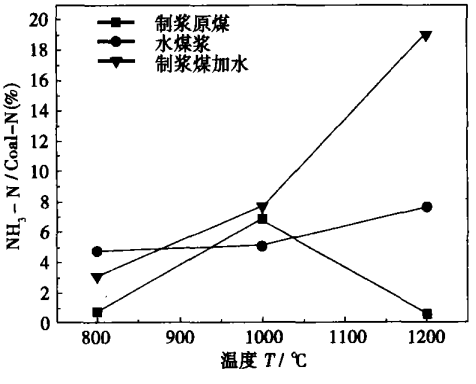

图 3　温度对 NH_3 释放的影响

随着温度的升高，水煤浆 NH_3 析出的量增加，虽变化缓慢，但是仍比制浆原煤释放出的 NH_3 量要多。原因可能是高温下，水分的存在一方面提供了 NH_3 形成所需要的大量的 H，另一方面由于它的扩孔作用提供了这些 H 自由基可吸附的焦表面。在焦的表面，H 自由基可以使含氮官能团活化，导致释放出更多的 NH_3，也正是由于提供了足够的 H 自由基，在 1 000 ℃以上，氮的氢化可以继续生成 NH_3。另外水煤浆中存在的大量钠离子可以促进 NH_3 的生成。

制浆原煤在水蒸气气氛下，随温度的升高，NH_3 的量一直呈增加趋势，而且温度到 1 000 ℃后，增加更加迅速，比制浆煤和水煤浆析出 NH_3 的量都多。原因可能是 HCN 和 NH_3 的生成都需要自由 H，而在煤的热解过程中，煤中碳氢链中的 N 基既可以与自由 H 结合生成 HCN 和 NH_3，也可以与碳氢链中的其它活性基团结合生成其它化学性质相对稳定的化合物。高温下水蒸气气氛可以提供大量的自由 H，使得 N 基能够及时与这些自由 H 碰撞，以 HCN 或 NH_3 的形式释放出来，因此大大促进了 HCN 和 NH_3 的生成。

3.3 焦中含氮量随温度变化的规律

图 4 为样品热解后焦中含氮量随温度变化的规律图。温度升高，样品的焦中含氮量成近线性下降，水煤浆热解时，析出的 HCN、NH_3 增加，水煤浆焦中的含氮量相对比较低，但比原煤在水蒸气气氛焦中的含氮量高。由于水煤浆中含有大量的水分，热解时水首先汽化为水蒸气，水蒸气可以提供大量的 H、OH 活性分子促进煤中的含氮官能团以 HCN 和 NH_3 的形式释放出来，水蒸气也会打开煤焦封闭的孔隙，使焦的表面积和孔容积变大，温度越高，扩孔作用越明显，这有助于减小挥发分氮析出的阻力，有利于挥发分的释放，从而其挥发分含氮量高，相应的留在煤焦里的含氮量减少。

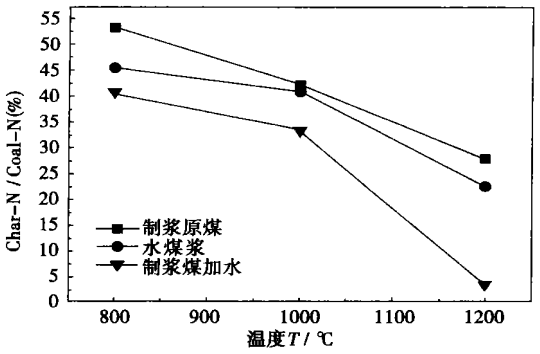

图 4 焦中含氮量随温度变化的规律

从图 2～图 4 中可以看出，原煤在水蒸气气氛下和水煤浆热解析出的 HCN、NH_3 都比原煤高，因为水蒸气的引入使得含氮官能团气化生成 HCN 和 NH_3 所需要的活性 H 的数量大大增加，同时水蒸气的引入也可能促使挥发分中的焦油进一步裂解生成 HCN 和 NH_3。而水煤浆与原煤在水蒸气气氛下析出量的不同可能是由于水蒸气可以提供的 H 自由基地数量不同，以及水煤浆在制备过程中，洗煤脱灰过程中去掉了大量的矿物质与加入添加剂过程中改变了煤中元素的成份及结构的原因。

尽管气氛中水分较大，能够使燃料尽可能大地释放出 HCN 和 NH_3，在还原气氛下对生成的 NO_x 进行还原，但是在实际燃烧过程中，如果水分过多，水分蒸发汽化时，需要吸收大量的汽化潜热，对于燃料的着火及燃烧均不利，故实际燃烧时要综合考虑，并非燃烧气氛中水分越多越好，显然水煤浆燃烧比煤粉单独燃烧以及煤粉喷水蒸气燃烧更好。

4 结 论

利用固定床反应器对水煤浆及其制浆原煤在惰性气体条件下、制浆原煤在水蒸气气氛进行高温热解试验；分析温度、水分对 HCN、NH_3 的释放影响，得出了以下结论：

（1）制浆原煤和水煤浆 HCN 的释放量，随温度增加变化缓慢，趋于稳定。而在水蒸气气氛下，HCN 的量随温度变化增加迅速，析出量远远超出煤及水煤浆的释放量。

（2）制浆原煤 NH_3 释放量随温度升高先增加后有下降，在 1 000 ℃左右出现一个析出峰值，温度继续升高，NH_3 的量不再增加反而略有降低；水煤浆 NH_3 析出的量随温度缓慢增加，但是仍比制浆原煤释放出的 NH_3 量要多；制浆原煤在水蒸气气氛下，随温度的升高，NH_3 的量一直呈增加趋势，温度到达 1 000 ℃后，增加更加迅速，释放量大于制浆煤和水煤浆的 NH_3 释放量。

（3）由于水蒸气可以提供的 H 自由基数量较大，故制浆原煤在水蒸气气氛下热解产生的 HCN 和 NH_3 的释放量最高，焦中含氮量最低；水煤浆与制浆

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由表 3 可知, 自然风干的 D1 号脱硫剂比表面积明显高于 105 ℃风干的 D2 号脱硫剂, 虽 D2 号的平均孔径比 D1 号大, 在脱硫反应初期其孔隙结构更利于 SO_2 和烟气中的水蒸气扩散进入脱硫剂的内部; 但随脱硫反应的进行, D2 号脱硫剂中的水分会很快被蒸发, 孔隙的表面附近的湿度逐渐降低, 且固硫产物会逐渐覆盖未反应的脱硫剂表面, 这些都会导致脱硫剂活性降低。而 D1 号脱硫剂由于内部水分含量高, 平均孔径较小, 使得脱硫剂孔隙的表面附近能长时间维持较高湿度; 同时水蒸气不断向外扩散, 破坏了覆盖的固硫产物层, 暴露出新鲜脱硫剂, 使得 SO_2 气体仍然能够较容易地扩散到脱硫剂内孔中, 这些因素使脱硫剂仍然具有较高的活性。表现在图 6 中为: 在脱硫开始的 14 min 内, D2 号脱硫剂的固硫效果好于 D1 号脱硫剂, 但在整个脱硫时间段内 D1 号脱硫剂表现出了更好的脱硫活性。

经蒸汽重整的 D3 号和 D4 号脱硫剂虽比表面积有了显著地增加, 但平均孔径减小, 内部孔隙结构变成以小孔为主, 使得在脱硫反应中, 更容易被脱硫产物堵塞孔隙, 从而降低了脱硫活性。因此, 蒸汽重整法不能使脱硫剂在保持较高含水量的同时也改善其孔隙分布。

4 结 论

考察了制备条件对添加剂调质钙基脱硫剂活性的影响, 实验发现, 在 70 ℃的水合温度下脱硫剂的固硫效果最好; NaOH 和 Na_2CO_3 的复合调质可使脱硫剂在较短水合时间内即表现出最佳的固硫效果; 自然风干方式制得的脱硫剂具有良好的孔隙结构, 脱硫活性高于烘干和蒸汽重整法条件下的脱硫剂。

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原煤相比 HCN 和 NH_3 的释放量要高, 而对应的焦氮含量要低。

(4) 综合考虑 HCN 和 NH_3 的释放量以及燃料的着火、燃烧, 显然水煤浆燃烧要优于煤粉的单独燃烧和煤粉喷水蒸气燃烧。

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(渠 源 编辑)

rous media under the condition of reciprocal flows. The solution under discussion comprises two constant differential equations, in which all control parameters have been included, thus contributing to an in-depth understanding of the effect of these control parameters on the characteristics of burners. Compared with the results of a numerical simulation, the temperature curves of porous-medium solids can be predicted exceedingly well by use of sectioned linear functions of the simplified solution. The maximal temperatures inside the burners obtained by making use of the simplified theoretical solution exhibit an identical tendency as that of the experimental values. However, the above maximal temperatures are usually greater than the experimental ones with the error between them being assessed at about 20%. **Key words**: theoretical solution, super-adiabatic combustion, porous medium, reciprocating

一种天然焦燃烧特性的试验研究＝**An Experimental Study on the Combustion Characteristics of a Kind of Natural Coke**[刊, 汉] /DONG Yong, WANG Chun-bing, WANG Wen-long, et al (Energy Source and Environment Research Institute Affiliated to Energy Source and Power Engineering College under the Shandong University, Jinan, China, Post Code: 250061) //Journal of Engineering for Thermal Energy &Power. — 2006, 21(4). —387～390

Natural coke is a kind of solid residue produced after coal has been heated and decomposed following its contact with magmatic rocks. It has been formed by destructive distillation after coal layers are subjected to heating and baked when magmatic rocks have intruded into coal layers or thereabouts. Natural coke is usually regarded as a kind of energy source difficult to be utilized. With a view to exploring new ways of comprehensive utilization of natural coke, an experimental study has been performed of such combustion characteristics as ignition and burn-up etc. of the natural coke and Jining-originated coal as well as a mixture of the two with the help of a thermogravimetry analytic method. The thermogravimetric test results show that the ignition temperature of natural coke is 876.3 K, regarded as the highest ignition temperature followed by a second highest specific to blended coals. Jining-originated coal is characterized by the lowest ignition temperature. The natural coke, however, has the shortest burn-up time, Jining-originated coal an intermediate one and the blended coals require the longest burn-up time. Summing up the experimental study and theoretical analysis, the authors conclude that natural coke-blended coal fuel can be used in power-plant boilers. The present research findings can provide a basis for employing natural coke as power plant fuels. **Key words**: natural coke, thermogravimetry, combustion characteristics, ignition, burn-up

燃煤飞灰粒度对比电阻影响机制的试验研究＝**An Experimental Study of the Mechanism Governing the Impact of the Size of Coal-fired Fly Ash Particle on Specific Resistance**[刊, 汉] /QI Li-qiang, YAN Wei-ping, YUAN Yong-tao (Environment Science and Engineering College under the North China University of Electric Power, Baoding, China, Post Code: 071003) //Journal of Engineering for Thermal Energy &Power. — 2006, 21(4). —391～394

After the ash samples have been collected from three domestic power plants and fully incinerated in a high temperature furnace, they were sifted by employing a mechanical sifting method into four categories of particle diameters, i.e. ＞145 μm, 90～154 μm, 45～90 μm and ＜45 μm. On a self-developed DR type high-pressure dust specific-resistance test rig, the specific resistance of fly ash of various particle diameters were determined. The test results show that superficial and volumetric electric conduction will jointly affect the specific resistance of fly ash and, furthermore, fine particles have a higher porosity. As a result, the fly ash with relatively small particle diameters has a higher peak value of specific resistance Moreover, before the latter reaches its peak value, the more coarse the ash samples, the higher their specific resistance. After the specific resistance has reached its peak value, however, the governing rule will evolve in an exactly opposite way. **Key words**: fly ash, ash incineration, particle diameter, specific resistance

水煤浆热解过程中 HCN 和 NH_3 释放特性的分析＝**An Analysis of HCN and NH_3 Release Characteristics of Coal-water Slurry in its Pyrolysis Process**[刊, 汉] /MENG De-run, ZHAO Xiang, ZHOU Jun-hu, et al (Education Ministry Key Laboratory on the Clean Utilization of Energy Resources and Environmental Engineering under the Zhejiang University, Hangzhou, Zhejiang, China, Post Code: 310027) //Journal of Engineering for Thermal Energy &Power. — 2006, 21(4). —394～396, 400

A pyrolysis test was performed of coal-water slurry and its raw coal in an inertial atmosphere and of its raw coal in a vapor atmosphere on a fixed bed reactor to study HCN and NH_3 release characteristics. The results show that with an increase in temperature the amount of HCN released from the raw coal and coal-water slurry changes slowly and finally tends to be constant. In the vapor atmosphere, however, the amount of HCN released from the raw coal will with a change in temper-

ature quickly increase and the amount being separated out far exceeds the amount released from the raw coal and coal-water slurry. The amount of NH_3 released from the raw coal will with an increase in temperature first increase and then decrease and a peak value will emerge at about 1 000 ℃. If the temperature continues to go up, the amount of NH_3 will not increase, but on the contrary begin to decrease. The amount of NH_3 separated out from the coal-water slurry will increase with an increase in temperature. Although the change proceeds slowly, the NH_3 amount released is still larger than that released from the raw coal. In the vapor atmosphere, with a rise in temperature, the amount of NH_3 all along exhibits a tendency to increase and after the temperature reaches 1 000 ℃, such an increase will go on still more rapidly with the amount released to be larger than that released from the raw coal and coal-water slurry. When the amount of HCN and NH_3 released as well as the ignition and combustion of fuel are taken into account in a comprehensive way, it can be clearly seen that the combustion of coal-water slurry is superior to that of pulverized coal alone and of pulverized-coal with steam injection. **Key words**: HCN, NH_3, pyrolysis, coal-water slurry

添加剂调质下脱硫剂活性影响因素的实验研究=**An Experimental Study of Various Factors Affecting the Activity of Desulfuration Agents Modified by Additives**[刊, 汉] /ZHANG Hu, TONG Hui-ling, DONG Shan-ning, et al (Education Ministry Key Laboratory on Thermal Sciences and Power Engineering under the TSinghua University, Beijng, China, Post Code: 100084) // Journal of Engineering for Thermal Energy & Power. — 2006, 21(4). —397 ~400

Through fixed bed experiments, the effect of different preparation conditions on the activity of additive-modified calcium-based desulfuration agents was investigated. It was found during the tests that at a hydration temperature of 70 ℃ the solubility of $Ca(OH)_2$ in the hydrate solution attains an optimum value, increasing the generated amount of desulfuration active substance in the hydrated product. Under the compound modification of NaOH and Na_2CO_3, the crystal in the product layer contains relatively more defects, thus promoting the diffusion of Ca^{2+} in the desulfuration agent to the product layer and gas phase and enabling the desulfuration agent exhibit its optimal sulfur retention effectiveness in a relatively short hydration time (2.5 hours). The desulfuration agent made in the natural drying mode has a relatively high water content. Through an analysis of pore structures it was found that the average pore diameter is relatively small and the pore surfaces can maintain a relatively high humidity for a long time, making its desulfuration activity higher than that of desulfuration agents obtained under the condition of baking and steam reforming process. **Key words**: desulfuration, additive, preparation, fixed bed

CO_2 活化 $CaCO_3$ 浆液对半干法烟气脱硫影响的实验研究=**An Experimental Study of the Effect of CO_2-activated $CaCO_3$ Slurry on Flue Gas Desulfuration by Using a Semi-dry Method**[刊, 汉] /ZHANG Li, WANG Jian-bao, LIU Yun-yi (Chemical Engineering School under the Shenyang Chemical Engineering College, Shenyang, China, Post Code: 110142) // Journal of Engineering for Thermal Energy & Power. — 2006, 21(4). —401 ~404

$CaCO_3$ desulfuration agent slurry is activated by gas CO_2 to enhance the reaction activity of $CaCO_3$ with SO_2, thus achieving a higher efficiency of the flue gas desulfuration process based on a fluidized semi-dry method. Through experiments in a fluidized reactor with a height of 1.1 m and an inner diameter of 12.5 cm, the effect of such factors as activation time, saturation proximity, calcium-sulfur ratio and desulfuration agent particle diameter etc. on desulfuration efficiency is investigated with coarse sand having a diameter of 275 μm and motionless bed height of 98 mm to serve as the fluidized medium. The experimental results show that when calcium-sulfur ratio is 1.2, saturation proximity at 15 ~18 ℃ and desulfuration agent particle diameter 64 μm, the $CaCO_3$ desulfuration agent after being activated by CO_2 gas can attain a desulfuration efficiency of 92%, approaching that of $Ca(OH)_2$ under the same conditions. **Key words**: flue duct gas, flue gas desulfuration, fluidized bed, sulfur dioxide

火电机组先进控制与优化软件的设计与应用=**Design and Applications of Advanced-control and Optimization Software for Thermal Power Plants**[刊, 汉] / YANG Bing, SUN De-min, GONG Dai-wei (Automation Department, China National University of Science and Technology, Hefei, China, Post Code: 230027), HAO Wei-dong (Shandong Electric Power Research Institute, Jinan, China, Post Code: 250002) // Journal of Engineering for Thermal Energy & Power. — 2006, 21(4). —405 ~408

To improve the control effectiveness of thermal power plants in China and enhance their operational efficiency, the authors have developed a set of advanced-control and optimization software through the adoption of an object-oriented technology