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中温下热解对半焦燃烧反应性的影响

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摘 要:用热天平(TGA)和粉末 X 射线衍射方法(XRD)分别测量了两种低变质程度烟煤和一种无烟煤的半焦在 400~1 400 ℃热解过程中燃烧反应性及其结构的变化,探讨了低变质煤的反应性变化的原因。研究发现,半焦反应性下降主要与热解过程中半焦晶格化与矿物质催化作用的逐渐消失有关,温度低于 900 ℃,原煤脱去大部分挥发份形成的半焦进一步热解时,晶格化现象不很显著,但反应性明显下降,反应性的降低主要与煤中矿物质在热解过程中的失活有关。

关 键 词:半焦;热解;燃烧反应性;晶格化;矿物质催化作用

中图分类号:TQ533 文献标识码:A

1 引 言

煤粉燃烧条件下,煤粉颗粒进入炉膛之后,其升温速率很高,达 10^4 ~10^6 ℃/s,大部分挥发份在几毫秒内释放完毕,然后为热解半焦燃烧,持续大约 1~3 s,这是煤粉燃烧的主要过程。某些条件下挥发份的释放与半焦的燃烧同时进行[1]。在动力扩散控制或扩散控制区的燃烧过程,半焦颗粒内部未燃烧部分受到热解。许多研究文献报导了煤粉炉燃烧条件下煤快速热解过程中反应性的降低[2~5]。反应性的变化似乎不能完全从孔隙和比表面积的角度来解释。对快速热解中半焦反应性的降低现象的分析表明,半焦反应性随热处理的强度如热解终温和停留时间等的增大而降低[2~6],但对半焦反应性的降低的原因有不同的认识。一种看法认为反应性的降低是由于半焦乱层炭结构的有序化导致了活性点的减少或降级[8~9];也有人认为加热条件下半焦的晶格化使得半焦炭网边缘的高活性碳原子对基面内的非活性原子的数量比降低,因而使半焦的反应性降低[4]。Davis 和 Hurt 的研究发现,煤粉炉飞灰中残碳的反应性的降低与半焦微晶参数的趋于石墨化有明显的关联[10~11],这在很多研究者的工作中得到进一步证实[2,4];还有一种观点认为矿物质的催化作用与半焦反应性的降低有关[12~13]。逐渐一致的观点是,快速热解中引起的反应性降低的主要原因是半焦中乱层炭结构有序化和半焦中弥散的含 Ca、Na、K、Fe 等元素的矿物质的催化作用的消失[14]。

半焦燃烧反应性的变化机理对煤燃烧模型的建立和飞灰含碳量的预测都非常重要。本文以两种低变质烟煤和一种无烟煤为例,模拟循环流化床锅炉中的热解与燃烧条件,温度范围为 900~1400 ℃,停留时间较长,研究分析半焦反应性的变化和半焦乱层炭结构有序化及矿物质催化作用之间的关系。

2 实验方法

2.1 半焦的制备

除传质和流动外,循环流化床锅炉中的燃烧条件的主要特征是炉内温度比煤粉炉低,在 850~900 ℃之间;入炉煤粒径大,筛分宽。由于在中温下燃烧而且颗粒粒径大,燃烧反应受动力扩散控制,而且大部分颗粒的停留时间比煤粉炉长得多,所以本文实验采取管式炉热解,以保持较长的停留时间。

管式炉以硅碳管作为加热元件,用可控硅自动控温。加热温度可恒定在设定值,变化不超过 2 ℃。

半焦的制备过程如下:将原煤磨至 180 μm,然后将研磨后的原煤粉送至管式炉内,在惰性气氛中 900 ℃下脱挥发份 7 min;将在 900 ℃下脱挥发份 7 min得到的半焦再送至管式炉在不同条件下热解,称为热处理。热处理温度 t_{HT} 为 400~1 400 ℃,热处理时间 τ_{HT} 为 4~24 h。

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2.2 反应性测量

半焦的燃烧反应活性(以下简称反应性)用 Du Pont 951 热天平(TGA)测量,采用恒温方法。样品 5.0 mg,均匀摊平在样品盘中,天平内通高纯氮气流并加热至 500 ℃,停留数分钟达到热平衡后,再切换成空气,氮气和空气流量都保持在 150 ml/min。以样品 500 ℃下与氧气反应过程中的最大失重速率 R_{500} 作为其燃烧反应性[7]:

$$R_{500} = -\frac{1}{w_0}\left(\frac{dw}{d\tau}\right)_{max} \quad t = 500 \text{ ℃} \tag{1}$$

式中:w_0—样品初重;mg;$(dw/d\tau)_{max}$—最大失重速率,mg/min。

实验条件保证了反应在动力控制区内进行,减小了颗粒粒径、空气流量的变化对反应性测量的影响。重复性实验表明,反应前热天平温度可稳定在 500±1 ℃,R_{500} 相对误差为 ±5%。

2.3 XRD 测量

煤的 X 射线衍射图非常弥散,表明它是一种无定形物质,是非晶质炭,在微小尺度下有类似于石墨,但并不与之完全相同的碳网结构。描述非晶质炭结构的理论有 Debye 和 Scherrer 提出的微晶理论以及 Warren 提出的乱层炭结构模型等[15]。只有高煤化程度的煤的 X 射线图谱才出现和石墨相近的较明显的(002)峰以及较矮的(100)、(004)和(110)峰。不同原煤脱挥发份后形成的半焦炭结构非常相似,XRD 图谱都出现(002)峰,但在较大 Bragg 角位置的三维晶格衍射,(100)、(004)和(110)并不出现,只出现二维的(10)峰。可利用 Bragg 方程和 Scherrer 方程计算微晶参数,如从(002)峰的位置和峰宽计算出微晶芳香层层间距 d_{002} 和芳香层堆垛高度 L_c,从(10)峰的峰宽可算出平均微晶直径 L_a[16]。

在热处理过程中,逐渐提高温度,乱层炭结构间断地趋向不同有序状态。在 500 ℃以下半焦原有的基本的六元碳网芳香结构变化甚微,在 800~1 500 ℃之间,这些基本芳香层面对面堆垛排列,但各堆垛之间是扭曲的,1 600~2 000 ℃之间,相邻堆垛开始联合形成波纹层状结构,2 100 ℃以上,波纹变平,晶体结构变得完善,最后变成石墨[17]。所以即使在煤粉炉燃烧条件下颗粒温度最高达到 1 600~1 800 ℃,乱层炭结构仍不能变成石墨,Hurt 称这个条件下乱层炭结构有序化过程为前期石墨化[11]。Franklin 用微晶理论来解释乱层炭结构有序化过程,认为对易石墨化炭,石墨化或晶格化指两相邻粗糙定向的微晶逐渐移动而使其 C 轴相平行,最后合并长成更大的微晶[15]。这种说法虽不十分妥当,但为简单起见,本文对低于 1 400 ℃下热处理过程中半焦乱层炭结构有序化现象仍用晶格化这一术语来描述。

由于半焦的 XRD 衍射图与一般晶体的很不一样,不好直接从原始衍射图谱上测出(002)和(10)峰的半高宽,本文采用 Short 方法先对原始 XRD 数据进行处理,测出(002)峰的位置、峰宽和(10)峰宽,再用有关公式计算 d_{002}、L_c 和 L_a[16]。

本文实验使用 Rigaku Ru300 X 射线衍射仪,最大功率为 18 kW,采用高速旋转铜阳级,K_a = 1.5406A。狭缝系统 1.0°(DS)-1.0°(SS)-0.60 mm(RS);电压 60 kV,电流 300 mA;连续扫描;计数间隔 $\Delta(2\theta)$ = 0.040°/p,计数时间 1.60 s/p;扫描范围 2θ = 5°~70°。

3 实验结果与分析

本文研究的煤种为两种低变质程度烟煤和一种无烟煤,分别为蔚县烟煤(YX)、湔江烟煤(JJ)和柳江无烟煤(LJ),3 种原煤的元素分析见表 1,工业分析见表 2。

表 1 煤的元素分析 (%)

	LJ	YX	JJ
碳	84.63	75.01	80.96
氢	2.53	4.32	5.30
氮	1.15	0.88	1.84
硫	1.47	1.20	0.66
氧	10.23	18.58	11.23

表 2 煤的工业分析析 (%)

	LJ	YX	JJ
水份(干基)	3.48	9.47	1.35
灰份(干基)	53.99	21.84	44.94
挥发份(干燥无灰基)	13.83	37.61	41.07
固定碳(干燥无灰基)	86.17	62.39	58.93
热值/MJ·kg^{-1}	12.41	19.70	17.27

3.1 半焦反应性的变化

半焦反应性随热处理的强度增加而减小,见图 1 和图 2。热处理时间超过某一临界值,反应性不再变化,出现一渐近值。热处理温度 t_{HT} 越高,渐近值

越低，临界时间越短。对 JJ 半焦，热处理温度 t_{HT} 升到 1 400 ℃后，R_{500} 几乎下降到零，即热处理后的半焦在 500 ℃下与氧气反应速度很慢。用压汞仪测量 900 ℃下不同热处理时间获得半焦的比表面积，结果表明，脱挥发份后半焦在热处理过程中比表面积并无明显变化，至少不足以解释图 1 和图 2 中反应性的大幅度下降。无烟煤 LJ 的反应性很低，其原煤的反应性 R_{500} 小于 1 %/min，热处理后半焦在 500 ℃下几乎不反应。

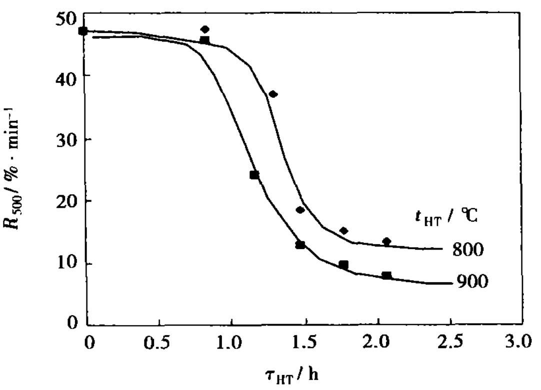

图 1　YX 半焦反应性随热处理时间的变化

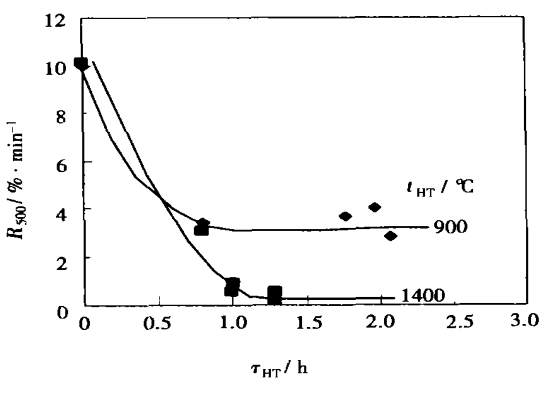

图 2　JJ 半焦反应性随热处理时间的变化

循环流化床燃烧温度通常控制在 850 ~900 ℃，但在燃烧时，半焦颗粒温度会大大超过床温[18]，所以某些煤形成的半焦如 JJ 半焦在此低温条件下燃烧时，会存在较明显的晶格化现象，并导致反应性的明显下降。

3.2　半焦结构的变化

将脱挥发份后半焦在某一温度下热解足够长时间，直到经 TGA 检测反应性不再明显变化，然后取出做 XRD 测量，得到的 d_{002} 和 L_c 随热处理温度 t_{HT} 的变化情况（见图 3 和图 4）。

从图 3 和图 4 可以看出，在 800 ℃以下，YX 半焦的微晶芳香层层间距 d_{002} 基本不变化，900 ℃时有一个突然降低，乱层炭结构跃迁到一个更有序的状态。此后，即使 t_{HT} 升至 1 400 ℃，d_{002} 并不进一步减小。与 YX 相反，JJ 半焦的 d_{002} 随热处理温度 t_{HT} 的升高呈连续下降趋势，若继续提高 t_{HT}，d_{002} 将趋向石墨的晶格层间距 $d_{002} = 3.3756 \times 10^{-10}$ m。图 4 说明随停留时间的增加，半焦中微晶有所长大，表现为芳香层堆垛高度 L_c 的增大。

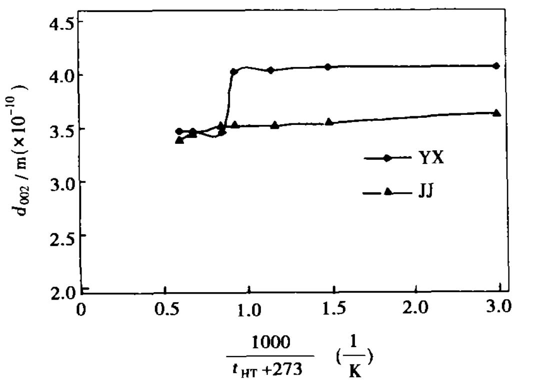

图 3　d_{002}　随热处理温度 t_{HT} 的变化

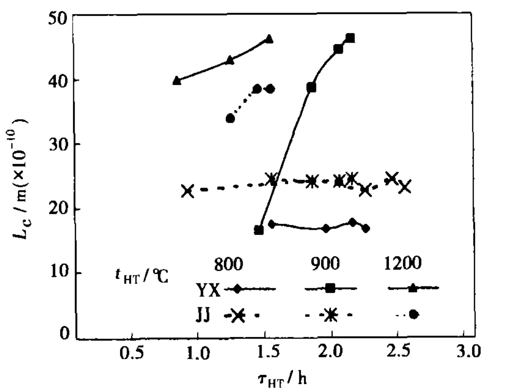

图 4　芳香层堆积高度 L_c 随热处理时间的变化

对 LJ 半焦的 XRD 测量发现，L_c 随热解温度的增加稍有增大，但 d_{002} 基本不变化，因为 LJ 是无烟煤，变质程度高，在中低温下碳结构基本不变化，反应性的变化很小。

3.3　矿物质催化作用

为考察矿物质的催化作用，参考 Radovic 的方法

将原煤酸洗除去矿物质后[4]，测量煤的反应性，结果见表3。YX 煤被除去矿物质后，反应性大大降低，接近在 900 ℃热解2 h 后的半焦的反应性。而 JJ 煤经酸洗后反应性虽然也降低了，但不很大，而且要明显比脱挥发份后半焦及 900 ℃热解 120 min 后半焦的反应性大。

表 3 原煤、酸洗原煤及半焦的反应性比较(R_{500}, %/min)

	A	B	C	D
YX	43.6	12.0	47.1	7.48
JJ	10.0	7.7	3.21	2.69

A—原煤；B—酸洗原煤；C—900 ℃下热解焦炭；D—热处理时间 2 h 的焦炭

从图 1 可以看出，因为 900 ℃以下热解半焦不发生明显晶格化，反应性变化主要是矿物质失活引起，且矿物质催化作用的消失较缓慢，在热解过程中持续 1 h 以上。不同热解温度 t_{HT} 对应不同的反应性渐近值，有可能是因为经历较长的停留时间后矿物质的活性已基本失去，而某一 t_{HT} 下半焦炭结构达到某个有序状态后将不再变化，加之如前所述的半焦孔结构和比表面积在热解中改变甚小，这时反应性也就不变而固定在某个值。

4 结 论

(1) 热解过程中半焦燃烧反应性的变化与半焦的晶格化和矿物质催化作用的失去有关；

(2) 在中低温热解过程中，两种低变质烟煤的半焦反应性将连续降低至一渐近值，温度越高，反应性渐近值越低，而无烟煤的反应性变化不大；

(3) 对 YX 煤，900 ℃以下热解过程中半焦的晶格化不明显，失去矿物质的催化作用十分显著，半焦反应性的降低主要是矿物质催化作用的失去所导致；

(4) JJ 煤易于石墨化，随热解温度的升高，晶格化较明显，脱挥发份后半焦在 900 ℃以下热解过程中反应性变化和晶格化均不显著，而矿物质催化作用不大；

(5) 虽然循环流化床的燃烧温度通常控制在 850～900 ℃之间，但在燃烧时，半焦颗粒温度会大大超过床温，所以某些煤形成的半焦在炉内燃烧时，存在较明显的晶格化现象，并导致反应性的明显下降。

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composite fluidization mode, which can adapt to boiler load variation. Moreover, a PDA (Phase Doppler Anemometer) measurement system was employed to test the gas-solid two-phase flow field under this kind of fluidization mode. As a result, the gas-solid tangential speed and concentration distribution were obtained under the condition of a change in swirl airflow and an imaginary tangential radius in the circulating fluidized bed. Test results indicate that the tangential velocity in the composite-fluidization circulating fluidized bed will increase with an increase in radius and the gas-solid tangential slip speed is higher than that of the straight flow fluidization. There will also be an increase in concentration in the desulfurizer and a higher intensity in internal circulation, resulting in a corresponding increase in desulfurizaion efficiency.
Key words: circulating fluidized bed, composite fluidization, tangential velocity, internal circulation

旋涡式低温煤粉燃烧器的实验及其数值研究＝**Experimental and Numerical Study of a Swirl-type Low-temperature Pulverized-coal Burner** [刊,汉] /ZHU Bo, WANG Xiao-han, YAN Chang-feng, et al (Guangzhou Institute of Energy Conversion under the Chinese Academy of Sciences, Guangzhou, China, Post Code: 510640) //Journal of Engineering for Thermal Energy & Power. — 2005, 20(5). —501～505

Based on the principle and flow field organization of air staged combustion aimed at reducing NO_x emissions the authors have designed a swirl-type low-temperature pulverized-coal burner featuring low NO_x emissions. By employing an experimental and numerical method a study was conducted of its flow field characteristics. The results of the study indicate that there is at the lower portion of the burner an intensive swirl flow zone, which can contribute to the formation of a reduction and burn-out zone required by the grading of air. As regards the primary and secondary air feeding location and mode there exists for the flow field organization an optimized proportioning option. The numerical simulation of a two-phase flow motion in the burner based on a RNG $k-\varepsilon$ model has reproduced the experimental results of the flow field, reflecting the motion law of particulates of various sizes in a zoned gas-flow field. **Key words**: swirl-type burner, coal combustion, air staged combustion, low NOx emissions

基于小波变换的气化燃烧状态诊断试验研究＝**Experimental Investigation of the Diagnosis of Gasified Combustion Status of a Wavelet-based Transformation** [刊,汉] / LIANG Qin-feng, YU Guang-suo, NIU Miao-ren, et al (Research Institute of Clean Coal Technology under the East China University of Science & Technology, Shanghai, Post Code: 200237) //Journal of Engineering for Thermal Energy & Power. — 2005, 20(5). —506～508, 516

An effective method is proposed for analyzing through pressure signals the flame gasified combustion condition of an entrained flow gasifier. Under this method a scalar decomposition approach has been utilized to analyze furnace pressure time-domain signals. A study has revealed that in a cold state prior to ignition there exists no characteristics frequency. Under the condition of ignition and flame-extinction the pressure signals experience a step change with the signals at 0 Hz being subjected to a relatively great shock. In time of flame intensive fluctuations there exists a characteristic frequency of about 2 Hz. With the flame in a stable combustion state there will be a characteristic frequency of about 40 Hz. The above phenomenon indicates that within a certain range of frequencies the pressure signal distribution is closely related to the flame combustion condition in the gasified furnace. With the strengthening of flame combustion stability the pressure signals will shift in the direction of higher frequencies. **Key words**: wavelet transformation, entrained flow gasifier, flame, combustion diagnosis

中温下热解对半焦燃烧反应性的影响＝**The Impact of Medium-level Temperature Pyrolysis on Semicoke Combustion Reactivity** [刊,汉] / LIU Yan-xia, LU Jun-fu, LI Yong, et al (Department of Thermal Energy Engineering under the Tsinghua University, Beijing, China, Post Code: 100084) //Journal of Engineering for Thermal Energy & Power. — 2005, 20(5). —509～512

By employing the method of thermogravimetric analysis and powder X-ray diffraction measured respectively were the combustion reactivity and structural changes of the semicoke of two low-rank bituminous coals and one type of anthracite during the process of pyrolysis at 400～1400 ℃. The cause of the variation in reactivity of the low-rank coals was also discussed. A study has revealed that the lowering of semicoke reactivity is mainly due to the semicoke crystal-lattice growth and the gradual disappearance of catalytic action of minerals during the pyrolysis process. At a temperature lower than 900 ℃ during the further pyrolysis of the semicoke formed by raw coal being deprived of a majority of volatile matter the crystal-lattice growth phenomenon is not very significant. However, there emerged a marked reduction of reactivity, which is mainly related to the loss of activity of minerals in coal during the pyrolysis process. **Key words**: semioke, pyrolysis, combustion reactivity, crystal-lattice growth, catalytic action of minerals

不同粒径煤粉燃烧后一次颗粒物的特性研究＝**A Study of the Characteristics of Primary Particulate Matter after the Combustion of Pulverized Coal of Different Particulate Diameters** [刊，汉] /LU Jian-yi, LI Ding-kai (Key Laboratory for Thermal Energy Sciences and Power Engineering under the Tsinghua University, Beijing, China, Post Code: 100084) //Journal of Engineering for Thermal Energy & Power. — 2005, 20(5). —513～516

A sedimentation furnace was employed as a combustion device to study the characteristics of primary particulate matter formed after the burning of pulverized coal of different particulate diameters. Under an oxidizing atmosphere at 1100 ℃ pulverized coal of different particulate diameters was burned. With the help of an Andersen particulate striking device the particulate samples were separated and collected after combustion. It can be shown that the combustion of pulverized coal of relatively small particulate diameter can yield a greater quantity of fine particulates and produce a vast amount of submicron particulates. An analysis has revealed that this is caused by the different approaches whereby the particulate matter was produced. The results of the scanning of samples by an electronic microscope indicates that after the combustion of pulverized coal of two different particulate diameters the resulting products of approximately equal size may assume different appearances. Through an analysis the process of the formation of particulate matter following the burning of pulverized coal of two different particulate diameters was revealed. By utilizing the results thus obtained the authors have come up with a concept of environment-compatible pulverized coal fineness. **Key words**: pulverized coal combustion, sampling, particulate matter, formation mechanism, pulverized coal fineness

离心风机三维流场动力学特征和泄漏损失特性研究＝**A Study on the Dynamics Characteristics of the Three-dimensional Flow Field and the Leakage Loss Features of a Centrifugal Fan** [刊，汉] /LI Chun-xi, LEI Yong, WANG Song-ling, et al (Department of Power Engineering, North China University of Electric Power, Baoding, China, Post Code: 071003) //Journal of Engineering for Thermal Energy & Power. — 2005, 20(5). —517～520

The study of the internal structure of a centrifugal fan to raise its operating efficiency is of major significance for achieving energy savings and cost-effectiveness enhancement at a thermal power plant. On the basis of a standard $k-\varepsilon$ turbulent model and by using software Fluent a numerical simulation and analysis was conduced of the three-dimensional flow field in a model G4-73 No.8D centrifugal fan as well as in fans fitted respectively with vortex-prevention rings of type A and B. A study of the dynamics characteristics of the internal flow field and the clearance leakage of the fans indicate that compared with fans not being fitted with a vortex-prevention ring the flow field of fans additionally fitted with a vortex-prevention ring tends to be more uniform. With the effective crushing of large-sized vortexes the leakage loss has been drastically reduced. Moreover, in respect of large-sized vortex breakdown and the reduction of leakage losses the vortex-prevention ring of type B is markedly superior to that of type A. The research findings can serve as a theoretical basis for the energy savings-related modification of fans. **Key words**: centrifugal fan, flow field, vortex-prevention ring, leakage loss, numerical simulation

天然气炭黑燃烧特性的热天平研究＝**Thermogravimetric Study of the Combustion Characteristics of Natural-gas Soot** [刊，汉] /XIE Guang-lu, FAN Wei-dong, XU Bin, et al (Institute of Mechanical & Power Engineering under the Shanghai Jiaotong University, Shanghai, China, Post Code: 200240) //Journal of Engineering for Thermal Energy & Power. — 2005, 20(5). —521～526

By using a thermogravimetric analytical balance a study was conducted of the combustion performance of the soot obtained