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Paper

RESEARCH ON ULTRAFINE COMMINUTION OF MINERALS

BY THERMALLY ASSISTED HIGH PRESSURE WATER JET

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ABSTRACT

The paper put forward a new way for ultra-fine comminuting of mineral powders, that is, we combined high pressure water jet with heat for minerals ultrafine comminution. The mineral particles were damaged by thermal shock before comminuted, finally by high pressure water jet. With heated then quenched, the particles were shocked thermally and thermal stress was produced inside the particles. The mineral particles would produce lots of cracks or even be broken. On the one hand the effect of heating then quenching particles reduces greatly their strength and makes the latter comminuted easier. On the other hand the effects of high pressure water jet acting on the particles, such as impact, water wedge and cavitation, could ultimately achieve comminution for mineral particles. Experiments on the ultrafine comminution of kaolin, coal powder have been carried out with a device in our laboratory. The experimental results show that it is an effective way for particles ultrafine comminution.

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1. INTRODUCTION

Thermally assisted comminution is applying the general natural phenomenon of expand with heat and contract with cold of materiel to fracture or crack, that is, the local stress concentration when a material is heated or cooled the difference of expand and contract in single crystal of the material anisotropy or between adjacent crystals or the contract of materiel suddenly quenching [1-3]. On the one hand it can change mineral mechanical properties to make the mineral grind more easily. On the other hand it can accelerate mineral fracture along the boundary of crystal and separate the materiel especial the useful ingredient from the gangue to largely improve the purity of comminuting production [4-5].

High pressure water jet comminution technique is a method that the high pressure water jet with huge energy acts on the comminuted materiel in certain way and produces transient pressure on the cleavage plane or the cranny of materiel to comminute the materiel [6]. There are two benefits using high pressure water jet comminuting technology. One is water jet can comminute the materiel to ultra-fine-range in lower input power. The other is that the materials are comminuted to ultra-fine granules and the inner different ingredients can be better divided due to high-speed liquid penetrating through the crystal verge [7]. At the same time it has the advantages such as safety, saving energy and high efficiency. So this new wet ultrafine comminuting technology is increasingly attracting people's attention.

Researching on thermal and high pressure water jet comminuting technology deeply, the authors thought that if we combine the thermal treatment with the high pressure water jet comminution, their respectively advantages will be utilized and we can realize the good comminuting effect. Namely, first we can change mechanical properties of the material and make the material pre-damage by means of the thermally assisted comminution, and then realize ultra-fine comminution using high pressure water jet technology of high efficiency, low power consume and clear. So we can improve the size reduction of particles and purity of the powder productions efficiently.

2 EXPERIMENT

2.1 Experimental Method and Raw Materials

Figure 1 is the experimental schematic diagram of thermally assisted water jet comminuting apparatus. The experimental procedure is shown in figure 2. Its method and process are shown as follows: First the particles are heat treated through heating them in muffle furnace and quenching them in the slurry container, then pour the mixed slurry, uniformly mixed in certain proportion, into the high pressure container. Use stop valve to change the direction of liquid stream. Start up the high-pressure

pump and adjust the pressure to certain value by adjust-pressure operating valve. It can make the mixed slurry inject from the nozzle and come into being high pressure water jet which acts the target to comminute the particles. In order to make the effect better, we can apply the repetitious and recurrent way that the comminuted slurry was put into the high pressure container to go through the comminuting process again. After sampling the comminuted particles, we check and analyze them though the SEM and TEM.

Coal is an important energy source, but it is also one of the dominant factors of environmental pollution. In order to benefit the environment and reduce the pollution, it is no time to delay to develop the cleaning coal technology. As the focal point of cleaning coal technology, the slurry of coal is thought of as a substitute of fuel for oil. The main raw material of it is super fine coal powder and water. If the coal can be comminuted to granularity of the slurry needed using the high pressure water jet, the slurrying technology will be simplified greatly. The coal is a kind of non-homogeneous substance. Its basic structural unit is anisotropic. It has the foundation of internal factor to occur crack and flaw by heating and quenching.

The super-fine-calcined kaolin is an important non-metal mineral produce. It is applied extensively in fields of industry, such as making paper, cable, paint and so on. Especially in the top grade copperplate paper, its amount is to 40%. In the technology of producing ultrafine calcined kaolin there are two all-important processes, that is ultrafine comminution and calcinations. If we can combined the calcining technological with thermally assisted comminution, that is, we can combined the technology of producing with thermally assisted high pressure water jet comminution, we can realize the purpose of high efficiency and saving energy. In addition, the kaolin is a hexagonal crystalloid of samdwich which belongs under the anisotropic substance. In the process of expand with heat and contract with cold it is easy to destroy because the expand coefficients are different in different axial direction.

So we choose coal particles of less than 0.5mm and 325 mesh kaolin particles as the experimental materiel.

2.2 Experimental Results and Analysis

2.2.1 Comminution of Raw Coal Particles

One of the experimental materials is the Datong coal particles whose granularity is less than 0.5mm through screening. Some coal particles are heated to 350°C and then preserved heat for half an hour to make their temperature homogenous in the muffle furnace, then after they are launched into water of 16°C (the room temperature) to cool immediately and then comminuted by high-pressure water jet. By way of contrast some coal particles are comminuted directly by high-pressure water jet. The structure of the nozzle has been shown in the figure 3. Its diameter is 1.5mm, distance of target is 40mm and the ratio of solid and water is 45%. After comminuted, they were sampled respectively. Then we test and analyze the granularity through the SEM and compare the results of comminution under the two different conditions. The results of test are shown in the sheet 1.

From the table 1 we can see that whether the raw materials have been heat-treated or not, they can be comminuted through the high pressure water jet. The working pressure of comminution, the materials which were been heat-treated or not and times of cycle will affect the comminuted effect in certain degree. As for the comminuted effect, the coal particles which were heat-treated are better than the ones that weren't heat-treated.

If we want to get the equal granularity in the above two conditions, the working pressure of water jet will be reduced largely through heat treatment. With increasing of the working pressure, the effect of comminution will be improved. For coal particles after heat treatment, if the working pressure of water jet is above 20MPa, the content of the product whose granularity is less than $25\mu m$, will exceed 50%. If the working pressure of water jet is to 30MPa, the content of the product whose granularity is less than 45µm will be nearly 80% through once comminution and above 90% through two times comminution, and the granularity of the product is all less than 90µm through two times comminution. As for the material which was not heat-treated, the times of comminution will notably influence the effect of comminution. And to the material which was heat-treated, the first two times of comminution will remarkably influence the comminuting effect. But when the comminuting times are more than two, the influence is not remarkable. At the same time, when comminuting heat-treated and untreated coal particles in the same conditions we find that the former is narrower than the latter in the size distribution of products. Namely, the products of the former concentrate in the zone of small granularity. On the country the granularity of the latter is decentralization.

The SEM photograph of coal particles before comminution is figure 4. Figure 5 and figure6 are the SEM photographs of two states of coal after comminution at the same working condition of water jet, that is at 30MPa working pressure and the third comminution. The contrasting photographs indicate that coal particles can be indeed comminuted by high pressure water jet and the products of comminuting coal is finer and more uniform by the thermally assisted high pressure water jet comminution.

After heat-treated, the surface of the coal particle would occur the crack which can be observed by SEM and is shown in figure.7. But we couldn't observe the crack on the surface of the coal particle of untreated. It is proved that thermal treatment can produce the crack on the surface of the particle. Thermal treatment increased the crack which reduced the strength of particle and accordingly it made the comminution more easily.

2.2.2 Comminution of Raw Kaolin Particles

Another raw material is the 325 mesh kaolin particles, some of which heated in muffle furnace to 1150°C to reach the calcining temperature, held at that temperature for half an hour, quenched immediately in the water at room temperature of 16°C and then comminuted by the high-pressure water jet, another of which comminuted directly by high-pressure water jet.

The nozzle structure of water jet comminution shown in figure 3, the diameter is 1.0mm, target distance is 40mm, the ratio of solid and water is 35%. After comminution, we sampled and measured the granularity of products respectively by TEM and then compared and analyzed them. The results of measurement are shown table 2.

From table 2, we can see that the percentage of small granularity attained by comminuting heat-treated kaolin is higher than that untreated. Namely, the effect of thermally assisted water jet comminuting kaolin is good. At the same time, the working pressure of water jet affects notably the comminuting effect. The higher is pressure, the better is comminuting effect. And if products of the same granularity are gained, the working pressure of water jet will decrease largely by thermal treatment.

The experimental indicated that if heat-treated kaolin is comminuted by high-pressure water jet, high comminuting efficiency can be got in the first comminution above the certain pressure (e.g. 30MPa) and the efficiency increase obviously in the second but it is not so obviously in the third. However, when the working pressure is lower, with the increase of comminuting times the comminuting effect is improved obviously. As for the percentage of small granularity is produced by high pressure water jet comminution, untreated kaolin is far lower than heat-treated kaolin on the same water jet pressure, but the comminuting times would affect the comminuting effect of untreated kaolin at a certain extent. At the same time, the particle size distribution of comminution after heat-treating is narrower than that untreated.

The SEM photograph of raw kaolin particles before comminution is figure.8. It is shown that size of particles isn't uniformity. Figure.9 is the TEM photograph of the heat-treated kaolin particles at 10MPa working pressure after the first comminution and figure.10 is the TEM photograph of the untreated kaolin particles at 40MPa working pressure after the third comminution. From these photos we can find that the high pressure water jet can comminute kaolin particles and the heat-treated kaolin particles are comminuted more effective than the untreated them. Figure 11 is TEM photograph of surface cracks after heat-treatment for raw kaolin particles and the surface cracks are how comminution effect is perfect. However with pressure and times increasing the products are finer and the powder would come into being agglomeration.

3 CONCLUSION

This paper puts forward the way combining thermal comminution with high-pressure water jet to come into being thermal assisted high-pressure water jet ultrafine comminution. The cracks occur on the particle surface due to thermal treatment. Great deals of the cracks reduce the particle strength and make the comminution more easily. Based on a great lot of comminuting experiments, as well as measurement and analysis by means of SEM and TEM, the following conclusions can be drawn.

(1)Whether the kaolin particles are heat-treated or not, they can be comminuted using water jet. The water jet comminution has powerful ultrafine comminuting capacity.

(2) We can get good comminuting effect making use of thermal assisted high pressure water jet comminuting coal and kaolin. And when the pressure increases to a certain value, we can get high comminuting efficiency at the first comminution by the method.

(3) The comminuting times affect better the comminuting effect for the untreated material. On the country, the effect of the comminuting times is not so obvious for the heat-treated material. The particle size distribution of products by water jet comminuting heat-treated material is narrower and the particle size is more uniformity than untreated. At the same time, heat treatment can decrease largely the working pressure of water jet if the same granularity is gotten.

(4)The observations under the SEM indicate that thermal treatment can occur crack on the surface of particles. This is the main reason why the comminuting effect is so evident applying the thermal assisted water jet comminution.

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Pressure	State of	Commin	Particle Size x (µm)					
(MPa)	Material	-uting	x≤10	10< x ≤25	25< x ≤45	$45 < x \le 90$	x >90	
		Times	(%)	(%)	(%)	(%)	(%)	
10	Untreated	1	2.15	10.68	18.97	26.56	41.64	
		2	3.32	11.46	23.73	27.64	33.85	
		3	3.96	12.60	25.47	27.14	30.83	
	Heat-treated	1	11.18	18.36	25.98	25.33	19.15	
		2	21.15	24.63	22.81	21.18	10.23	
		3	29.86	23.19	20.69	18.83	7.43	
20	Untreated	1	5.27	13.79	22.65	26.43	31.86	
		2	10.51	17.77	23.50	25.43	22.79	
		3	21.96	21.60	20.47	20.14	15.83	
	Heat-treated	1	23.67	24.15	20.39	18.97	12.82	
		2	35.89	27.43	19.87	10.35	6.46	
		3	37.77	28.94	19.19	9.39	4.71	
30	Untreated	1	14.13	18.41	23.02	25.23	19.21	
		2	20.15	21.16	22.84	20.18	15.67	
		3	28.86	24.19	18.67	15.83	12.45	
	Heat-treated	1	44.67	28.98	15.42	8.74	2.19	
		2	55.26	29.17	9.50	6.07	0	
		3	62.06	21.92	10.18	5.94	0	

Table1. The Particle Size Distribution of the Comminuted Coal

Pressure (MPa)	Sstate of Material	Commin -uting Times	Particle Size x (µm)					
			x≤2	$2 \le x \le 5$	$5 \le x \le 10$	$10 < x \le 20$	$20 < x \le 45$	
			(%)	(%)	(%)	(%)	(%)	
10	Untreated	1	34.74	21.66	18.91	14.63	10.06	
		2	35.90	22.42	19.23	13.19	9.26	
		3	37.17	21.41	19.82	12.67	8.93	
	Heat- treated	1	45.84	18.33	16.36	12.35	7.12	
		2	48.23	19.91	16.86	10.57	4.43	
		3	52.23	18.93	16.76	8.15	3.93	
20	Untreated	1	42.56	18.24	16.71	12.63	9.86	
		2	44.83	19.12	16.21	12.15	7.69	
		3	46.13	20.11	15.42	11.61	6.73	
	Heat- treated	1	55.98	19.70	11.83	6.55	5.94	
		2	63.29	18.20	10.76	5.32	2.43	
		3	66.40	16.32	11.15	4.16	1.97	
30	Untreated	1	55.51	18.12	11.34	8.49	6.54	
		2	61.73	19.01	8.46	6.58	4.22	
		3	65.26	17.82	8.89	5.62	2.41	
	Heat- treated	1	71.29	13.46	9.36	3.81	2.08	
		2	73.43	16.07	7.15	2.32	1.03	
		3	73.50	17.12	7.32	1.78	0.28	
40	Untreated	1	65.07	16.02	8.96	5.62	4.33	
		2	67.65	15.57	8.34	5.16	3.28	
		3	70.13	15.65	7.74	4.33	2.15	
	Heat- treated	1	76.88	11.69	7.35	3.02	1.06	
		2	78.95	12.81	7.04	1.20	0	
		3	79.68	13.19	5.51	1.62	0	

Table2. The Particle Size Distribution of the Comminuted Kaolin



Figure 1. Schematic Illustration of the Experimental Apparatus(1)High Pressure Pump;(2) Pressure Gauge; (3)Operating Valve;(4) Switching Valve; (5) High Pressure Vessel;(6)Slurry Pump;

(7)Slurry Container ; (8)Heater ; (9) Nozzle; (10-) Collect Chamber; 11-Target



Figure 2. Experimental Flow Chart of Thermal Assisting Water Jet



Figure 3. Fluid Jet Nozzle



Figure 4. SEM Photograph of Raw Coal Particles



Figure 5. SEM Photograph of Comminution Products for Untreated Coal



Figure 6. SEM Photograph of Comminution Products for Heat-treated Coal



Figure 8. SEM Photograph of Raw Kaolin Particles before Comminution



Figure 7. The Cracks on the Surface of Heat-treated Coal



Figure 9. TEM Photograph of First Comminution Products for Heat-treated Kaolin at 10MPa



Figure 10. TEM Photograph of Third Comminution Products for Untreated Kaolin at 40MPa



Figure 11. TEM Photograph of Cracks on the Surface of Heat-treated Kaolin Particle