



Self-heating tendency evaluation of sulfide ores based on nonlinear multi-parameters fusion

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Abstract: In order to reveal the nonlinear dynamics characteristics of unsteady self-heating process of sulfide ores, nine different kinds of sulfide ore samples from a pyrite mine in China were taken as experimental materials and their self-heating characteristics were measured in laboratory. Furthermore, the measured temperature was studied by integrating wavelet transform, nonlinear characteristic parameters extraction and fuzzy comprehensive evaluation. The results indicate that only the ore samples 1, 2, 6 and 9 have obvious self-heating phenomenon, and their self-heating initiative temperatures are 220 °C, 239 °C, 220 °C and 220 °C, respectively, which means that they are difficult to produce self-heating under normal mining conditions. The correlation dimension of self-heating process is fraction and the maximum Lyapunov exponent is positive, which means that it is feasible to study the self-heating process based on chaotic dynamics theory. The nonlinearities of self-heating process of these four samples (ore samples 1, 2, 6 and 9) are 0.8227, 0.7521, 0.9401 and 0.8827 respectively and the order of the samples according to these results is: sample 6, sample 9, sample 1, sample 2, which is consistent with the measured results of self-heating characteristics. Therefore, the nonlinearity method can be used to evaluate the self-heating tendency of sulfide ores, and it is an effective verification of the reliability of measured results.

Key words: sulfide ores; self-heating process; nonlinear characteristic parameter; nonlinearity; self-heating tendency

1 Introduction

Spontaneous combustion of sulfide ores is a conversion of the chemical energy to heat due to oxidation [1–3]. If the reaction heat doesn't dissipate entirely, sulfide ores will be heated and release more and more heat, which is a positive feedback. When the temperature of sulfide ores reaches the ignition temperature, spontaneous combustion will take place. It is well known that spontaneous combustion of sulfide ores is one of the most serious disasters in sulfide deposits mining [4–6]. According to statistics of spontaneous combustion, it has occurred in more than ten mines in China since 1949, such as Wushan Copper Mine, Xiangshan Pyrite Mine and Dongguashan Copper Mine [7]. Spontaneous fires of sulfide ores not only generate massive toxic gas and heat to worsen the work

environment, but also bring about large economic losses even loss of human life. For the rapid decrease of surface mineral resources, deep mining has become a trend and its high temperature can cause spontaneous combustion more easily. Therefore, researches on the oxidation mechanism, prevention and control technology of spontaneous combustion are a premise to ensure safe and efficient mining for high-sulfur mines.

In recent years, evaluation for spontaneous combustion tendency of sulfide ores has attracted attention of many researchers and some new evaluation methods have been proposed, such as apparent activation energy method [8], matter-element model [9], evaluation model based on entropy and set pair analysis theory [10], Fisher discriminant analysis method [11] and uncertainty measurement model [12]. To evaluate the spontaneous combustion tendency of sulfide ores synthetically, self-heating initiative temperature, which is measured by

carrying out self-heating characteristics experiment, is commonly used as an index. As the self-heating of sulfide ores is a nonlinear multi-factor coupling evolution process, research on the unsteady self-heating process by means of nonlinear dynamics theory is worthy of further study. Currently, there are very scarce researches on nonlinear characteristics of self-heating process.

In this work, nine kinds of typical sulfide ore samples from a pyrite mine in China were taken as experimental materials and their self-heating characteristics were measured with precision instruments in laboratory. Combined with wavelet transform, nonlinear characteristic parameters extraction and fuzzy comprehensive evaluation, the nonlinearity of ore samples self-heating process, which is the characterization of complexity for self-heating process, was calculated. Moreover, self-heating tendency evaluation method of sulfide ores based on nonlinear multi-parameters fusion was proposed.

2 Experimental

2.1 Ore samples collection and analysis

Sulfide ore samples were collected from a pyrite mine in China with the sampling method of multi-point sampling, and nine types of representative samples were taken as experimental materials. As an example, the chemical composition of the ore sample 1 is listed in Table 1.

Table 1 Chemical composition of ore sample 1 (mass fraction, %)

Water-soluble iron ion	Total sulfur	Monomer sulfur	Sulfate ion
0.0016	35.27	0.044	1.57

The metallic mineral of the ore sample 1 is pyrite with two-stage mineralization. The largest size of early-stage pyrite particles is about 5 mm, and their average size is about 2 mm. They are fragmented as a result of the stress action. Unlike the early-stage pyrite particles, the late-stage pyrite particles are fine-grained aggregates with the average size of about 5 μm . The micrograph of the ore sample 1 is shown in Fig. 1.

2.2 Experimental method

In the experiment, the particle diameter of ore samples was ground to less than 0.2 mm. Each sample with mass of about 100 g and moisture content of about 5% was installed into the reactor in the automatic heating incubator to measure its self-heating characteristics owing to the weak oxidation of sulfide ores at normal temperature. The initial temperature was set between 40

and 50 °C and one single heating extent was about 10 °C. After the temperature of automatic heating incubator reached the setting temperature, it was kept isothermal for about 30 min. During the experiment process, the maximum temperature of automatic heating incubator was not higher than 250 °C. The experimental devices are shown in Fig. 2.

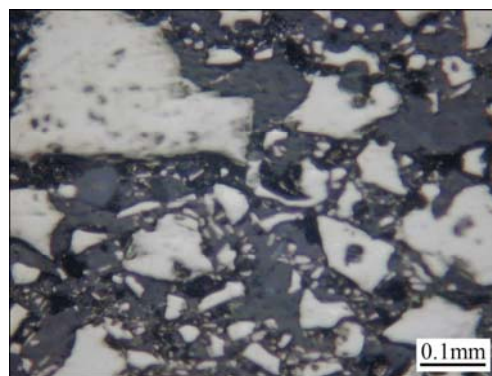


Fig. 1 Micrograph of ore sample 1

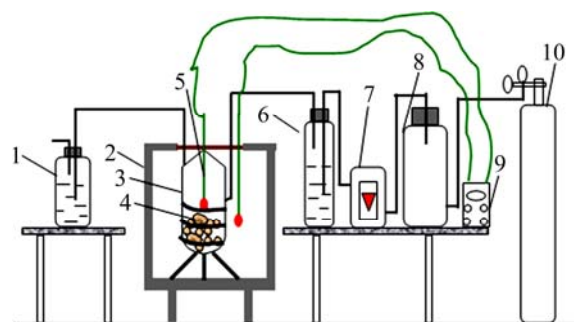


Fig. 2 Schematic diagram of experimental apparatus: 1—Poisonous gas absorption bottle; 2—Automatic heating incubator; 3—Reactor; 4—Ores; 5—Temperature probe; 6—Humidifier; 7—Flow meter; 8—Buffering bottle; 9—Automatic temperature recorder; 10—Oxygen cylinder

3 Experimental results

By analyzing the self-heating characteristic curves of ore samples, it can be found that only the ore samples 1, 2, 6 and 9 have obvious self-heating phenomenon, and their self-heating initiative temperatures are 220 °C, 239 °C, 220 °C and 220 °C, respectively, which means that they are difficult to produce self-heating under normal mining conditions. Figure 3 displays the self-heating characteristic curve of the ore sample 1. It shows that the obvious self-heating period lies between 1200 and 1230 min and the maximum self-heating extent is 5 °C.

4 Preprocessing of measured temperature series

Temperature variations of ore samples are caused by the comprehensive effect of automatic heating incubator

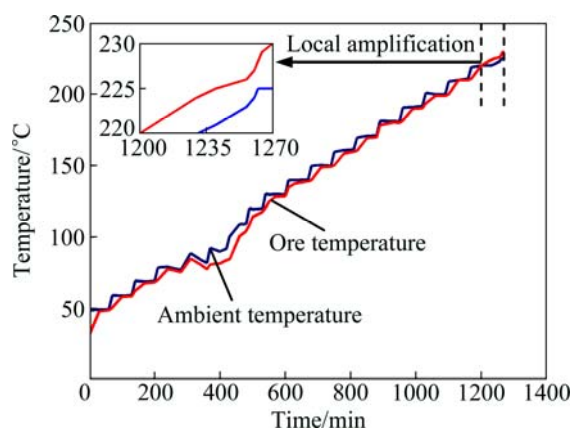


Fig. 3 Self-heating characteristic curve of ore sample 1

and exothermic oxidation of ore samples, and the former plays a dominant role. Hence, it is necessary to separate self-heating information from the measured temperature series. Then, the temperature series were processed by wavelet decomposition and reconstruction [13]. The low frequency components of the series (large scale approximation parts) reflect the effect of gradient temperature-elevating with automatic heating incubator, and high frequency components (details) contain the complicated self-heating information of ore samples.

Owing to the limited measured data, the temperature series of the four samples (ore samples 1, 2, 6 and 9) were extended with the cubic spline interpolation method, and the length of the expanded series was 301. For temperature increments could show the pre- and post-temperature variations more intuitively, they were selected as the research objects.

The temperature increment series were transformed with the same wavelet function of bior3.1 based on the results of wavelet optimization. The characteristic values of corresponding series are listed in Table 2, which indicate that the temperature increment series are well correlated for all the autocorrelation coefficients, greater than 0.86, and the skewness coefficients have a significant difference.

For these four kinds of ore samples, their temperature increment series were decomposed with wavelet bior3.1, and then the first layer of high frequency coefficients were reconstructed. Finally, the

reconstructed high frequency series were normalized as the research series.

Wavelet reconstruction for temperature increment series of the ore sample 1 is shown in Fig. 4. The trend of low frequency reconstructed series, which reflects the effect of gradient temperature-elevating, is consistent with that of the original sequence in general. High frequency reconstructed series have more complicated detailed change, which can amplify the tiny distinction of self-heating process effectively.

5 Extraction of nonlinear multi-parameters

According to the definitions of approximate entropy, correlation dimension and the maximum Lyapunov exponent, each of them can be adopted to characterize the chaotic features from different aspects. Approximate entropy mainly reflects the disorder degree of the time series. Correlation dimension is a measure of the complexity for chaotic series. Maximum Lyapunov exponent is an estimation of predictability for chaotic series. Therefore, these three kinds of nonlinear characteristic parameters were extracted from research series as evaluation indexes for self-heating tendency.

5.1 Extraction of approximate entropy

For a given N -point research series $\{x(i), i=1, \dots, N\}$, approximate entropy can be obtained by the following steps (m is the dimension, and r is the threshold value) [14]:

1) Compose the m -dimensional vector $X(i)$ as follows:

$$X(i) = [x(i), x(i+1), \dots, x(i+m-1)],$$

$$i = 1, 2, \dots, (N-m+1) \quad (1)$$

2) For every i value, calculate the distance between $X(i)$ and $X(j)$, namely

$$d[X(i), X(j)] = \max_{k=0-(m-1)} [|x(i+k) - x(j+k)|] \quad (2)$$

3) Given the threshold value r ($r > 0$), for each i value, count the number of $d[X(i), X(j)] < r$ (denoted as $n_i^m(r)$) and calculate the ratio of $n_i^m(r)$ to the total number of vectors (denoted as $C_i^m(r)$), so

Table 2 Characteristic values for corresponding series of ore samples

Sample No.	Temperature increment series				Low frequency reconstructed series			
	Mean value	Variation coefficient	Autocorrelation coefficient	Skewness coefficient	Mean value	Variation coefficient	Autocorrelation coefficient	Skewness coefficient
1	0.6617	1.3556	0.8837	0.7875	0.6617	1.3462	0.8856	0.7510
2	0.6987	1.6351	0.8813	0.5807	0.6987	1.6149	0.8849	0.5218
6	0.8060	1.3528	0.8929	2.5053	0.8060	1.3401	0.8952	2.4664
9	0.7500	1.4255	0.8673	1.8876	0.7500	1.3964	0.8756	1.8521

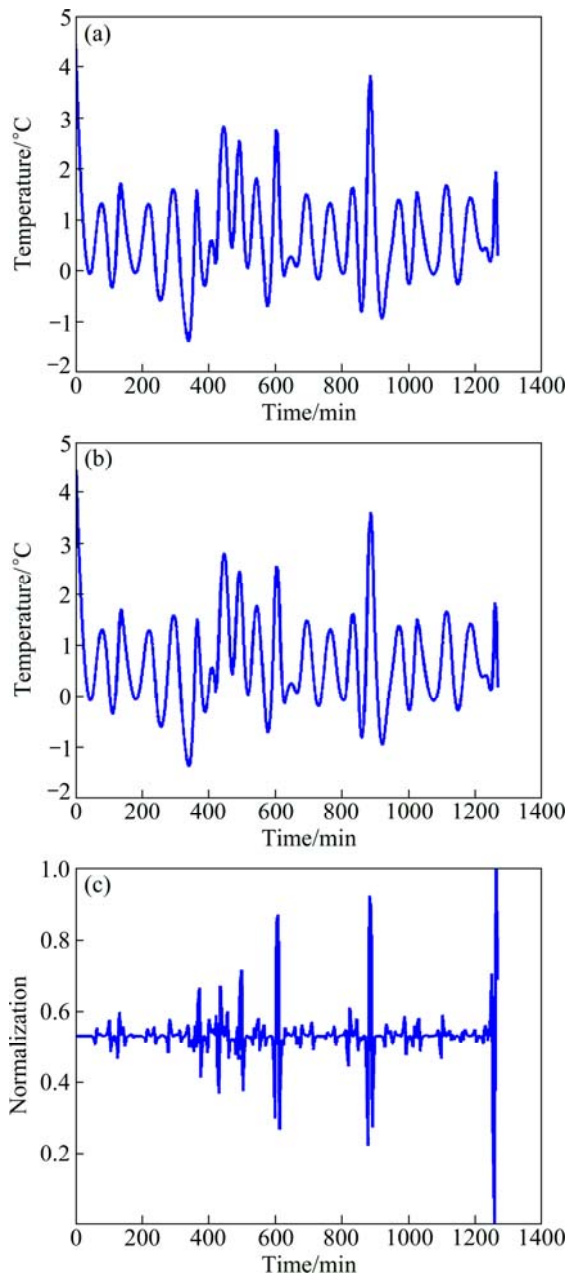


Fig. 4 Wavelet reconstruction for temperature increment series of ore sample 1: (a) Original series; (b) Low frequency series; (c) Normalized high frequency series

$$C_i^m(r) = \frac{n_i^m(r)}{N - m + 1} \quad (3)$$

4) Calculate the logarithm of $C_i^m(r)$ and its average value, denoted as $\phi^m(r)$, namely

$$\phi^m(r) = \frac{1}{N - m + 1} \sum_{i=1}^{N-m+1} \ln C_i^m(r) \quad (4)$$

5) For $m + 1$, repeat steps 1–4 to obtain $\phi^{m+1}(r)$. The approximate entropy value is as follows:

$$E_A(m, r) = \phi^m(r) - \phi^{m+1}(r) \quad (5)$$

Usually, $m=2$ and $r=k\sigma$ (k is empirical coefficient, $k=0.1-0.2$; σ is standard deviation of research series) are used to calculate the approximate entropy.

In this work, empirical coefficient was determined according to the variation extent of approximate entropy with different k values, which can be expressed as

$$E = \frac{|E_A(m, k_2\sigma) - E_A(m, k_1\sigma)|}{E_A(m, k_1\sigma)} \quad (6)$$

For all the ore samples, when the average E takes on minimum value, the corresponding k value is optimal.

Figure 5 denotes the variation of approximate entropy with different values of k . With the increase of k , approximate entropy for research series of the ore sample 9 gradually increases, while the others decrease, as shown in Fig. 5. When $k=0.15$, the average E takes a minimum value of 0.66%. Correspondingly, approximate entropies of the ore samples 1, 2, 6 and 9 are 0.6309, 0.6229, 0.5759 and 0.6251, respectively.

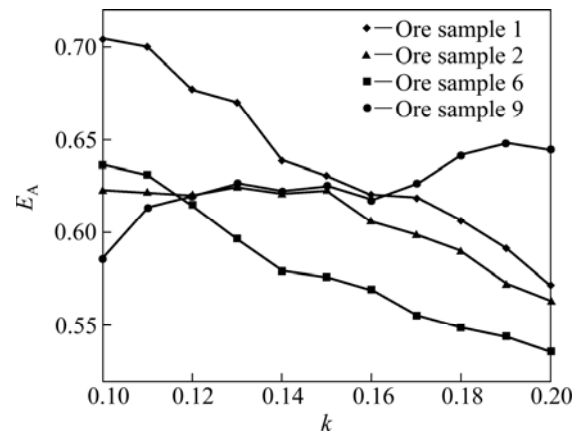


Fig. 5 E_A - k curves for ore samples

5.2 Extraction of correlation dimension

Correlation dimension can be calculated by the following steps based on the GP algorithm [15]:

1) For the m -dimensional reconstructed phase space, define the correlation integral as

$$C(m, r) = \frac{1}{N^2} \sum_{\substack{i, j=1 \\ i \neq j}}^N \Theta[r - |X_i - X_j|] \quad (7)$$

where N is the number of phase point, r is hypersphere radius, $|X_i - X_j|$ is the euclidean distance between the phase point X_i and X_j , and Θ is called Heaviside function.

2) Adjust the value of r in a certain range to meet

$$C(m, r) \propto r^{D(m)} \quad (8)$$

where $D(m)$ is an estimation of correlation dimension.

3) When the embedding dimension m is equal to the saturated embedding dimension m_c , $D(m)$ will be stable and the stable value is correlation dimension D_2 .

In actual calculation of D_2 , the optional delay time τ was obtained using the improved auto-correlation function method [16] firstly, and then D_2 was extracted in reconstructed phase space.

In $C(m,r)$ – $\ln r$ curves for research series of the ore sample 1 are depicted in Fig. 6(a), which indicate that $\ln C(m,r)$ increases at first and then tends to be stable with $\ln r$. The calculated results are $D_2=1.4503$ and $m_c=6$, as shown in Fig. 6(b).

The calculated results D_2 for all the samples are listed in Table 3.

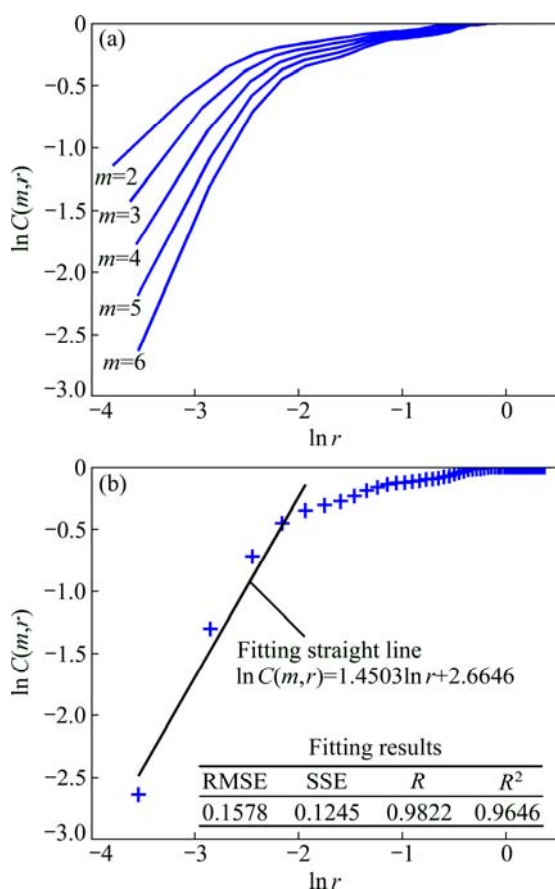


Fig. 6 D_2 calculation process for ore sample 1: (a) $\ln C(m,r)$ – $\ln r$ curves; (b) Fitting result ($m_c=6$)

Table 3 Calculated results D_2 for ore samples

Samples No.	τ	m_c	D_2
1	1	6	1.4503
2	1	6	1.1154
6	1	7	1.9921
9	1	6	1.5642

5.3 Extraction of maximum Lyapunov exponent

Maximum Lyapunov exponent can be calculated by the following steps by means of small data sets [17].

1) In the m -dimensional reconstructed phase space, search the nearest neighbor point X_{jj} of the reference

point X_j and keep temporal separation:

$$d_j(0) = \min \|X_j - X_{jj}\|, |j - jj| > p \quad (9)$$

where $d_j(0)$ is the initial distance between the phase point X_j and its nearest neighbor point X_{jj} and p is the mean period of research series.

2) To each point X_j , calculate the distance $d_j(i)$:

$$d_j(i) = \|X_{j+i} - X_{jj+i}\|, i = 1, 2, \dots, \min(N-j, N-jj) \quad (10)$$

3) For general j , calculate the average value of $y(i)$:

$$y(i) = \frac{1}{q\Delta t} \sum_{j=1}^q \ln d_j(i) \quad (11)$$

where q is the number of all non-zero $d_j(i)$.

4) Select the linear region of $y(i)$ – i curves and make the regression line using the least square method. The slope of the line is the maximum Lyapunov exponent λ_{\max} .

For the ore sample 1, the mean period of research series was calculated with the value of 3 based on FFT method. The phase space was reconstructed with $m=6$ and $\tau=1$, and then the maximum Lyapunov exponent ($\lambda_{\max}=0.0037$) was calculated following the above step. Figure 7 shows the λ_{\max} result for the ore sample 1.

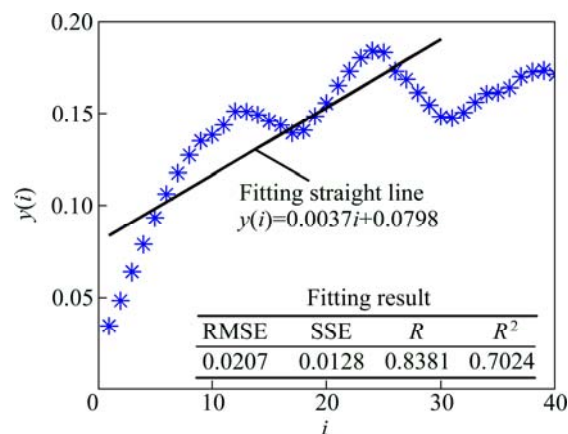


Fig. 7 λ_{\max} result for ore sample 1

Table 4 lists the calculated results of λ_{\max} for all samples. In conjunction with Table 3, it is known that for each sample, the correlation dimension of self-heating process is fraction and the maximum Lyapunov exponent

Table 4 Calculated results λ_{\max} for ore samples

Samples No.	p	λ_{\max}
1	3	0.0037
2	2	0.0019
6	3	0.0131
9	2	0.0072

is positive, which means that self-heating of sulfide ores is a chaotic evolution process. As a result, it is feasible to study the self-heating process based on chaotic dynamics theory.

6 Self-heating tendency evaluation by means of nonlinearity

The maximum Lyapunov exponent can be used to characterize the nonlinear degree of self-heating process, but it needs high precision long series to obtain the precise value and is sensitive to noise. So it is limited in actual use. To compensate for this deficiency, approximate entropy, correlation dimension and the maximum Lyapunov exponent were fused, and the integration value was defined as nonlinearity to be used to measure the degree of irregularity and complexity for the self-heating process from dynamic aspect.

According to the conception of nonlinearity, define the nonlinearity vector of self-heating process as $U=(u_1, u_2, u_3)$, where u_1, u_2 and u_3 are approximate entropy, correlation dimension and the maximum Lyapunov exponent, respectively. To eliminate their physical and geometric meaning and difference of order of magnitude, u_i ($i=1, 2, 3$) need to be normalized, namely $u_i \in U$, $U=(0, 1)$. Meanwhile, define the evaluation set as $V=(v_1, v_2, v_3, v_4)$, where v_1, v_2, v_3 and v_4 are corresponding to the ore samples 1, 2, 6 and 9.

Given the factors set U and evaluation set V , $\alpha: U \rightarrow V$ is univariate evaluation function, so $f(\alpha(u_1), \alpha(u_2), \alpha(u_3))$ is the comprehensive evaluation of U [18]. Generally, comprehensive evaluation function involves two weight vectors, namely normalized weight vector and regularization weight vector.

In this work, the normalized weight vector was obtained with the value of (0.37, 0.31, 0.32), and the univariate evaluation matrix $R=(r_{ij})_{3 \times 4}$ was established as

$$R = \begin{bmatrix} 1 & 0.9873 & 0.9128 & 0.9908 \\ 0.7280 & 0.5599 & 1 & 0.7852 \\ 0.2824 & 0.1450 & 1 & 0.5496 \end{bmatrix} \quad (12)$$

Four kinds of evaluation functions were utilized to assess the nonlinear intensity of self-heating process.

When using the weighted average model, namely,

$$p_{1j} = \sum_{i=1}^3 w_i r_{ij} \quad (13)$$

where w_i is the weight vector elements and r_{ij} is the scalar of normalized factors.

The evaluation value is $P_1=(p_{11}, p_{12}, p_{13}, p_{14})=(0.6861, 0.5853, 0.9677, 0.7859)$.

When using the geometric mean model, namely,

$$p_{2j} = \prod_{i=1}^3 r_{ij}^{w_i} \quad (14)$$

The evaluation value is $P_2=(p_{21}, p_{22}, p_{23}, p_{24})=(0.6047, 0.4483, 0.9668, 0.7634)$.

When using the univariate decision model, namely,

$$p_{3j} = \bigvee_{i=1}^3 (w_i \wedge r_{ij}) \quad (15)$$

The evaluation value is $P_3=(p_{31}, p_{32}, p_{33}, p_{34})=(1, 0.9873, 0.9128, 0.9908)$.

When using the main factors prominent model, namely,

$$p_{4j} = \bigvee_{i=1}^3 (w_i \nabla r_{ij}) \quad (16)$$

The evaluation value is $P_4=(p_{41}, p_{42}, p_{43}, p_{44})=(1, 0.9873, 0.9128, 0.9908)$.

The above four kinds of evaluation values were taken as the initial evaluations, and then all of the initial evaluations were further evaluated. The final evaluation matrix R_p was constructed based on the initial evaluation P_i ($i=1, 2, 3, 4$), namely,

$$R_p = (P_1, P_2, P_3, P_4)^T \quad (17)$$

According to the weighted average function, the formula of nonlinearity can be written as

$$\alpha = \sum_{i=1}^4 \gamma_i \eta_{ij} \quad (18)$$

where α is the nonlinearity, γ_i is the weight coefficient with the value of 0.25 and $\eta_1-\eta_4$ are the column vectors of R_p .

The sort of self-heating tendency and nonlinearity of ore samples are listed in Table 5. From Table 5, it is

Table 5 Sort of self-heating tendency and α of ore samples

Sample No.	Sort of self-heating tendency			Sort of α	
	Self-heating initiative temperature/ °C	Maximum self-heating extent/ °C	Sort	α	Sort
1	220	5	3	0.8227	3
2	239	3	4	0.7521	4
6	220	26	1	0.9401	1
9	220	15	2	0.8827	2

found that the sort of nonlinearity is consistent with that of the self-heating tendency. Thus, the self-heating tendency of sulfide ores can be evaluated by means of nonlinearity method.

The main purpose for the self-heating characteristics experiment of sulfide ores is to reveal the relationship between self-heating effects and the ambient temperature of automatic heating incubator and determine the lowest ambient temperature that induces rapid oxidation of sulfide ores. Theoretically, as the ambient temperature rises, the oxidation heat liberation increases and meanwhile the condition of heat dissipation deteriorates. When the ambient temperature reaches the critical value, the amount of heat liberation will be greater than that of heat dissipation, and obvious self-heating will appear in the ores due to the effect of heat accumulation simultaneously. But during the actual measurement process, temperature of ore samples often does not exceed the ambient temperature owing to the low heat liberation and other restrictive factors. Hence, it is difficult to determine the self-heating initiative temperature of ore samples accurately. In addition, the difference of temperature elevation program has a certain impact on the measured results of self-heating characteristics. Thus, the reliability of the evaluation results based on only the measured values in laboratory needs to be further verified.

The nonlinearity proposed in this work was extracted from all the measured temperature data by integration of multiple nonlinear characteristic parameters, and the effect of automatic heating incubator was eliminated with the wavelet technology during the calculation process. Since the self-heating of sulfide ores is a typical nonlinear evolution process, it is more objective to evaluate self-heating tendency with the nonlinearity method. The practical application indicates that the evaluation results are of high distinction and can be used to verify the reliability of the measured results.

7 Conclusions

1) The measured results of self-heating characteristics indicate that only the ore samples 1, 2, 6 and 9 have obvious self-heating phenomenon, and their self-heating initiative temperatures are 220 °C, 239 °C, 220 °C and 220 °C, respectively. Namely, sulfide ores of the pyrite mine are difficult to produce self-heating under normal mining conditions.

2) The extracted correlation dimension is fraction and the extracted maximum Lyapunov exponent is positive, which means that self-heating of sulfide ores is a chaotic evolution process. So, it is feasible to study the self-heating process based on chaotic dynamics theory.

3) The self-heating process nonlinearity of these

four samples (ore samples 1, 2, 6 and 9) are 0.8227, 0.7521, 0.9401 and 0.8827, respectively and the order of the samples according to these results is: sample 6, sample 9, sample 1, sample 2, which is consistent with the measured results of self-heating characteristics. Therefore, self-heating tendency of sulfide ores can be evaluated by using the nonlinearity method, which is an effective verification of the reliability of measured results.

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基于非线性多参数融合的硫化矿石自热倾向性评价

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摘 要: 为揭示硫化矿石非稳态自热过程的非线性动力学特征, 以从某硫铁矿采集的 9 种硫化矿石样品为实验材料, 开展室内矿石自热特性实验。采用集成小波变换、非线性特征参数提取和模糊综合评判等方法对实测温度数据进行研究。结果表明: 仅矿样 1、2、6 和 9 出现明显自热, 自热起始温度分别为 220、239、220 和 220 °C, 在正常采矿条件下矿石不易产生自热; 矿样自热过程的关联维数均为分数, 最大 Lyapunov 指数均大于 0, 验证了基于混沌动力学理论研究自热过程的可行性; 矿样自热过程的非线性度分别为 0.8227、0.7521、0.9401 和 0.8827, 排序依次为矿样 6、矿样 9、矿样 1、矿样 2, 与自热特性测定结果一致。因此, 可采用非线性度方法来评价硫化矿石自热倾向性, 该方法可对实测结果的可靠性进行有效验证。

关键词: 硫化矿石; 自热过程; 非线性特征参数; 非线性度; 自热倾向性

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