



Research paper

Investigation on the changing law of magnetoelectric anomalies in variable temperature coal rocks

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ABSTRACT

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The changing law of magnetoelectric anomalies in the metamorphic process of coal rock bodies is of great significance to the prediction and study of fire in hidden coal fields. For this reason, this paper reveals the changing law of magnetoelectric anomalies in metamorphic coal rock bodies through the study of pyrolysis characteristics, microcrystalline structural changes, and physical phase transfer of the coal rock bodies, based on the four coal samples with different metamorphic degrees, and captures and elucidates the thermal gravity (TG) curves and micro-thermogravimetric (DTG) curves of the coal. Thermolysis behavior and characteristics. The results show that the variation rule of electrical anomalies during the metamorphic process is small at the beginning of the process, the dielectric properties of the coal samples show a significant increase from T_2 to T_3 , and the dielectric constant reaches its maximum value at the T_4 temperature point. The X-ray diffraction test was used to comprehensively analyze the microcrystalline morphological parameters of the coal before and after heating. The results showed that the high temperature induced a denser stacking of the carbonaceous layers, which strengthened the van der Waals forces between the layers, shortened the aromatic layer spacing, enhanced the electronic coupling between the layers, and increased the dielectric constant of the coal, resulting in an increase in the dielectric loss as well as the tangent value of the dielectric loss, and a blockage of the charge transport pathway. Based on X-ray diffraction data from four coal and seven rock samples with different degrees of metamorphism, in-depth analyses were performed using JADE to identify multiple phases in the coal and rock samples using the Rietveld refinement program, and semi-quantitative compositional assessments of the phases were performed. The results indicate that the conversion of iron minerals to more magnetic ferromagnetic compounds such as hematite (Fe_2O_3) and magnetite (Fe_3O_4) enhances the magnetization of the coal, while decomposition and oxidation reactions during the warming of the rocks alter their ferromagnetic compound type and content.

1. Introduction

Coal occupies a key position in the global energy pattern and is an important basic energy source for many countries. In recent years, as the demand for coal resources development continues to rise, the characteristic changes of coal rock body in variable temperature environment have become the focus of research. In-depth investigation of the changing law of magnetoelectric anomalies of variable-temperature coal rock bodies (i.e., the phenomenon of nonlinear response of magnetization and dielectric constant of coal rock bodies under temperature change) is not only conducive to in-depth insight into the evolution of

the internal structure of the coal rock bodies at various temperatures, but also the role of the decomposition of the organic constituents and the transformation of the minerals in pyrolysis on the magnetoelectric properties [1,2]; but also provides theoretical support for the prediction and study of hidden fire zones in order to reduce the risk of spontaneous combustion of the coalfield [3].

Coalfield fire zone is a kind of extremely destructive disaster phenomenon, which has serious negative impacts on the rational utilization of resources, ecological balance and stability, and the harmonious development of society [4]. However, research on coal pyrolysis process focuses on a single physical parameter or a specific coal type, and it is of

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practical significance to detect coalfield fire zones by various methods and enhance the effectiveness of coalfield fire prevention and extinguishing engineering [5]. Lei Guo et al. [6] found that the water content in the coal body is the main factor influencing the dielectric constant through the study of lignite coal in Yunnan province. Qingdong Wang et al. [7] obtained the temperature dependence of the dielectric constant of lignite was calculated by artificial neural network, and Yunpeng et al. [8] found that the increase in temperature has a significant effect on the pyrolysis process of coal. This is due to the fact that during pyrolysis, a series of physical and chemical changes occur with increasing temperature, which include decomposition of the coal molecular structure, release of volatiles, and formation of coke. Shuqin Lin et al. [9] found that the dielectric constant and resistivity have a significant dependence on the pyrolysis temperature by investigating the dielectric constant response mechanism in the pyrolysis process. Hongqing Zhu et al. [10] obtained the characteristic temperature points by warming and thermogravimetric (TG) experiments, and found that the complex relative permittivity of coal samples decreased with the increase of temperature. The above studies were limited to the overall trend of the complex relative permittivity and did not investigate the temperature dependence of the characteristic temperature points and electrical anomalies in the pyrolysis stage.

Ma Mengya [11] studied the changes of pyrite's structure, morphology, and crystal structure during pyrolysis and found that the phase transformation of pyrite affects the pyrolysis products of coal. Zhang Xinhai et al. [12] used the Gouet method to determine the magnetization rate of the samples, the results show that: coal at room temperature presents antimagnetism, when the temperature reaches 200 °C the magnetization rate becomes positive, and in the range of the fire its magnetization rate rises sharply, after the temperature reaches 500 °C, the rate of increase tends to flatten out. Zhao Yuyang et al. [13] on the study of pyrrhotite magnetization rate results show that the magnetization rate of the pyrite to be affected by the heat treatment temperature, decreasing at 600 °C and increasing rapidly after 850 °C. Youying Chen et al. [14] discussed the magnetic characteristics of the combustion products of coals with different degrees of metamorphism indicating that the burned coal samples contain a large number of ferromagnetic minerals. Although the previous studies can know that the temperature change will have an effect on the magnetic characteristics of coal rocks, they have not elucidated the effect of carbon structure reorganization and mineral phase transformation on the magnetic properties and the quantitative analysis of the dynamic transformation of ferromagnetic minerals.

On the issue of coal rock microcrystalline structure and typical correlation, most of the existing studies used qualitative description and lacked quantitative parametric analysis based on ray diffraction (XRD). For example, Haiyu Liu et al. [15] argued that the presence of minerals plays a catalytic role in coke structure, but did not reveal the effect of microcrystalline parameters on charge transport. Han Wenbin [16] found that the minerals inside the coal samples changed with increasing temperature to produce more paramagnetic substances, and that the temperature increase continued to destabilize the electrons outside the nucleus while affecting the ordering of the magnetic moments of the magnetic domains. Although the transient electromagnetic method can detect anomalies in the fire zone, the resolution of the test results is relatively low for localized high-temperature zones. Zhenlu Shao [17] used the natural potential method, resistivity tomography (ERT), and magnetic method to explore the fire zone of a coal mine, and comprehensively verified the accuracy and effectiveness of the three methods. The high-density electric method can clearly distinguish the location of the anomalous area, for the comprehensive management of coal mines as well as to find the location and scope of the waterlogged hollow area to provide an effective method [18,19], in the exploration of coal mineral resources, hidden fire area [20] prevention and control of transient electromagnetic method has a wide range of applications, and its numerical analysis is also a more mature method so as to provide technical

references and guidance for the future production work [21,22].

For the study of rocks overlying coal beds, Song Wujun et al. [23] used the high-density electric method to detect and analyze the detection results and found that during the process of burning in the fire zone, moisture migrated upward, resulting in the decrease of rock resistivity and the formation of low-resistance anomalies. Shaofei Wang et al. [24] found that the change of magnetization and chromaticity of the loess in the fire zone of the coal mine was related to the degree of transformation of iron compounds with the temperature. Li Jingwen [25] found that the magnetization of coal rock increases with increasing grain size and temperature through magnetic measurement experiments on coal rock. Shichang Chen et al. [26] showed that the dielectric constant of the rock increases with increasing water saturation and mineralization through measurement of natural sandstone samples. In the above studies, the electrical anomalies as well as the magnetization anomalies in the coal rock warming process are limited by the vague knowledge of the physical phase transformation mechanism in the local high temperature zone.

In this paper, four kinds of coal samples with different metamorphic degree and seven kinds of rock samples were taken as research objects, and pyrolysis characterization tests before and after high-temperature treatment, experiments on the microcrystalline structure of coal and physical phase evolution were carried out to systematically reveal the changing law of magnetic and electric anomalies of metamorphic coal and rock bodies. The dynamic correlation of dielectric and magnetic parameters at the characteristic temperature points($T_1 \sim T_4$) was analyzed by thermogravimetric(TG) and microtemperature-gravimetric (DTG) curves; the evolution of microcrystalline structural parameters was quantitatively analyzed based on XRD to analyze the change of dielectric parameters; and the phase transition paths of ferromagnetic minerals were semi-quantitatively tracked through the Rietveld refinement program, revealing the mechanism of chemical and structural coupling for the magnetization rate anomaly. The experimental results not only fill the gap of the theoretical multi-dimensional correlation of "pyrolysis-structure-phase", but also provide a new theoretical basis and application reference for the detection of coalfield fire zone and the prevention and control of mine fires.

2. Characterization of coal pyrolysis under different temperature gradients

2.1. Experimental sample selection and preparation

In this paper, four kinds of coal samples with different degrees of metamorphism, namely lignite, barren coal, gas coal and anthracite, and nine kinds of rock cores of the overlying rock strata of coal seams obtained from drilling were selected as the research objects in Shanxi, (the specific numbers and types of rock cores are shown in Table 1). The selected coal samples and cores were crushed using a pulverizer (e.g., Fig. 1) and then screened to produce a powder with a particle size range of 38–79 μm in a sealed bag for spare use (e.g., Fig. 2).

Table 1
Overburden core categories.

Numbered	Categories	Numbered	Categories
R1	Fine-grained sandstone	R6	Medium-grained sandstone
R2	Charcoal-bearing fine sandstone	R7	mudstone
R3	Fine-grained sandstone	R8	Muddy sandstone
R4	Sandy Mudstone	R9	Fine-grained sandstone
R5	Carbonaceous mudstone		



Fig. 1. Crusher.



Fig. 2. Coal rock powder 38–79 μm particle size.

2.2. Characterization of coal pyrolysis under different temperature gradients

Thermal analysis experiments using STA6000 comprehensive thermal analyzer (as shown in Fig. 3) will be prepared particle size 38–79 μm of the four kinds of coal samples were weighed out 10 ± 0.5 mg, placed in the crucible ready for testing. The instrument test conditions: initial temperature 30 °C, termination temperature 800 °C, the rate of



Fig. 3. STA6000 integrated thermal analyzer.

temperature increase of 10 °C / min, the test chamber into the helium gas as a protective gas, the ventilation volume of 50 ml / min. Thermogravimetric analyzer temperature accuracy and sensitivity of 0.1 °C and 0.1 μg , respectively.

2.3. Analysis and study of characteristic temperatures in pyrolysis processes

By plotting thermogravimetric (TG) and microtremor gravimetric (DTG) curves for four coal samples with different degrees of metamorphism (shown in Fig. 4). These curves critically capture the pyrolysis behavior and properties of the coal. The TG curves reflect the change in sample mass with increasing control temperature; the DTG curves are derived from the TG data and represent the change in the rate of sample mass loss, i.e., the rate of change in mass per unit of time, providing insight into the thermal stability and decomposition rate of the samples.

Characteristic temperature points occupy a key position in evaluating coal pyrolysis properties and show significant variations in TG and DTG curves, such as pyrolysis onset temperature, temperature corresponding to the maximum mass loss rate, and end decomposition temperature, etc. The TG curves provide a comprehensive quantitative analysis of the thermal decomposition process of the coal samples, whereas the DTG curves reveal more precisely the details of the dynamic changes of the mass loss during the decomposition process. In summary, the TG curve and the DTG curve together provide an important analytical tool for studying the thermal behavior of coal, its reaction mechanism and its thermal stability.

The values of the characteristic parameters related to coal pyrolysis were obtained from different coal pyrolysis TG and DTG curves during the warming process [27]. From the DTG curves, it can be seen that there are two obvious weight loss peaks T_1 and T_4 , the onset temperature of the main stage of pyrolysis T_3 and so on. The first weight loss peak T_1 is caused by the release of CO_2 and CH_4 gases due to the breakage of weakly bonded chains and functional groups in the coal samples, and it is also the cause of the physical adsorption of water and small molecule gases in the coal samples; the second weight loss peak T_4 is the maximum point of pyrolysis reaction intensity, and the intensity of pyrolysis reaction of the coal samples increases obviously with the increase of temperature; T_2 is the temperature point of the maximum loss rate of the DTG curve; T_3 is the intersection of the tangent line made at the level of the DG T_3 is the intersection of the tangent line at the horizontal point of the TG curve and the tangent line at the inflection point of the curve, and it is also the reference temperature point for the onset of the pyrolysis process. At this stage the macromolecular structure of the coal sample is broken and removed, and chains and functional groups with weak bonding energies are broken. A large amount of carbon dioxide-based gases, water, methane and short-chain aliphatic hydrocarbons are produced. As the temperature rises, the TG curve continues to fall, which can be regarded as the stage of condensation and polymerization of coal samples, and tar and hydrogen gas become the main products in this stage.

Combining the above analyses, the key performance indicators of pyrolysis were derived in this study with the help of detailed thermogravimetric analysis and summarized in a table (shown in Table 2). These characteristic parameters include the peak weight loss temperature points(T_1 , T_4),the maximum weight loss rate temperature point(T_2),and the pyrolysis reaction onset temperature point(T_3).The data provided in the table reflect in detail the dynamic characteristics of the mass change (mass loss rate of 25 %–38 %)and thermal stability performance of different coal samples during the heating process, which provides an empirical basis for further investigation of the changing law of electrical parameters in the process of coal pyrolysis.

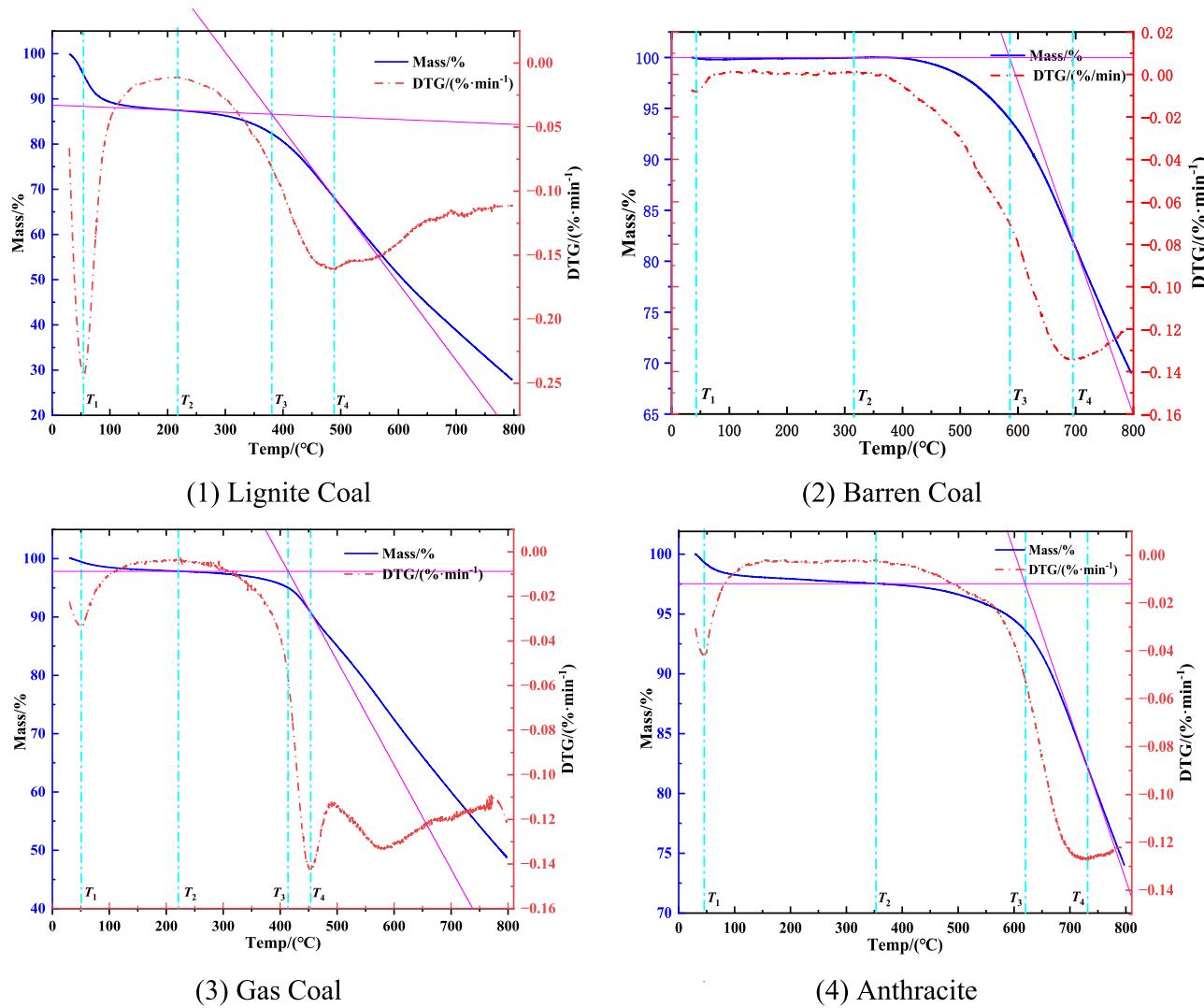


Fig. 4. Coal pyrolysis characteristic curve.

Table 2
Indicators of pyrolysis characteristic parameters of coal samples.

Coal Type	T_1 / °C	T_2 / °C	T_3 / °C	T_4 / °C
Lignite Coal	54.6	219.5	379.6	487.2
Barren Coal	42.2	317.4	561.2	699.2
Gas Coal	51.2	226.1	414.3	452.3
Anthracite	45.6	352.1	618.6	732.1

2.4. Response law of coal pyrolysis characteristics to magneto-electric anomalies

The characteristic temperature point of coal pyrolysis is one of the key parameters for evaluating its pyrolysis properties, and all stages of the pyrolysis process result in significant changes in the dielectric behavior of coal. At the initial stage of pyrolysis, the change in the dielectric properties of coal samples is relatively small, which is mainly affected by water evaporation. Entering into the T_2 to T_3 stage, as the temperature of the thermal decomposition reaction continues to rise, the thermal movement within the coal molecules becomes more and more active, and this active thermal movement leads to changes in the interaction between the coal molecules, so that the charge transport channel within the coal molecules is gradually broadened, and the charge transport capacity can be significantly improved. Because of this,

the dielectric properties of coal show a very obvious rising trend, when the thermal decomposition process advances to the T_4 temperature point when the thermal reaction of coal samples has basically arrived at the end point and the value of dielectric constant rises to the peak, reflecting that the electrical parameters of the coal samples have reached the maximum degree at this particular temperature.

Coal samples with different degrees of metamorphism have different effects on their resistivity. Lignite and gas coal with lower degrees of metamorphism have relatively high porosity due to their higher moisture content and more low molecular compounds mainly in chain structure, so their resistivity rises rapidly in this stage and reaches the maximum value in the stage of T_1 - T_2 from ambient to T_1 with the evaporation of the moisture of the coal samples with the rise of temperature. For the barren coal and anthracite with high degree of metamorphism, the resistivity of the three coal samples started to decrease rapidly from the initial stage of heating due to their relatively stable microcrystalline structure, which is difficult to be affected by the evaporation of water molecules. When the temperature is gradually increased to the starting temperature point T_3 of the pyrolysis reaction the thermal motion between coal molecules is intensified, and some carriers in the coal are activated thereby increasing the migration rate of electrons and ions. The pyrolysis reaction of coal is basically completed as the temperature increases to T_4 , when the concentration of carriers decreases accordingly, which in turn leads to a slowing down of the rate

of decrease in its resistivity. This process reflects the effect of the change of internal physical and chemical states on the electrical properties of coal with the change of temperature during the pyrolysis process. The enhancement of thermal motion and the activation and concentration change of carriers, together, act to change the resistivity of coal, showing a trend of rapid decline followed by a slowing down of the rate of decline.

In terms of the anomalous response of the magnetization rate during coal pyrolysis, the magnetization rate of coal did not show significant changes from room temperature to T_3 stage. This is mainly due to the fact that within this stage, the main processes occurring are the removal of moisture from the coal and the release of volatiles. And when the temperature reaches the vicinity of T_3 , with the increase of temperature and the further depth of pyrolysis, the carbon structure begins to undergo rearrangement, as well as the chemical change of the minerals, and these factors begin to have an effect on the magnetization rate. It is worth noting that carbon materials have the potential to enhance their own magnetic properties during the transformation process, whereas changes in the ferromagnetic minerals contained in coal during pyrolysis have a direct impact on the magnetization rate. Most of the previous studies focused on a single coal type or a single parameter (e.g., mass loss rate), and lacked the dynamic correlation between the characteristic temperature points ($T_1 \sim T_4$) and the magnetoelectric parameters. In this section, the TG/DTG curves are used to clarify the dynamics under the characteristic temperature points of the coal samples with different degrees of metamorphism, and to reveal the mechanism of the differentiation of the metamorphism degree on the electrical response. This correlation between the physical and chemical changes of coal and magnetization rate at different temperature stages is important for a deeper understanding of the evolution of magnetic characteristics during coal pyrolysis process.

3. Microcrystalline structure of coal under different temperature gradients

3.1. Coal rock X-ray diffraction testing

Four kinds of raw coal samples and coal samples after 700 °C heat treatment were tested by using a D8ADVANCE X-ray diffraction instrument from Bruker, Germany. Powder samples of seven overburden rocks were selected for testing under variable temperature *in situ* conditions. The instrument X-ray tube voltage is 40 kV, current is 30 mA, detection range (2θ) is 5–90°, scanning speed is 10°/min, anode target material is Cu target, K α radiation, X-ray wavelength is 0.15406 nm, and angular reproducibility is 0.0001 degrees.

3.2. XRD experimental investigation of the microcrystalline structure of coal

Weng Chengmin et al. [28] pointed out that the transition state between crystalline and amorphous coal body structure is called microcrystalline structure in their study of the Peak Coalfield, which provides an important theoretical basis and methodological guidance for subsequent in-depth research on the microcrystalline structure of coal. The microcrystalline structure of coal refers to the orderly arrangement of aromatic layers in the carbon skeleton, which is a transitional structure between the completely disordered amorphous state and the highly ordered graphite crystals. The parameters of the structure are the crystal plane spacing d_{002} , the stacking degree L_c , the ductility L_a , the average number of aromatic layers N_{ave} , and the degree of graphitization g . They can be obtained by the Braggs and Scherrer formula:

$$d_{002} = \frac{\lambda}{2\sin\theta_{002}} \quad (1)$$

$$L_c = \frac{0.89\lambda}{\beta_{002}\cos\theta_{002}} \quad (2)$$

$$L_a = \frac{\infty 1.84\lambda}{\beta_{100}\cos\theta_{100}} \quad (3)$$

$$N_{ave} = \frac{L_c}{d_{002}} + 1 \quad (4)$$

$$g = \frac{a_1 - d_{002}}{a_1 - a_2} \quad (5)$$

Note: λ is the X-ray wavelength 0.15406 nm; θ_{002} is the diffraction angle of the 002 peak, °; β_{002} is the width of the 002 peak, nm; θ_{100} is the diffraction angle of the 100 peak, °; β_{100} is the width of the 100 peak, nm; a_1 is the maximum directional layer spacing of the initial state, 0.3975 nm; and a_2 is the facet spacing of the perfect graphite crystal, 0.3354 nm.

In order to obtain the characteristic parameters of the morphological and structural features of the microcrystalline structure of the coal, the peak 002 and peak 100 of the coal samples need to be fitted and analyzed separately, and the split-peak fitting is implemented by the built-in Peak Fitting Analysis Module of Origin2023. In the specific operation process, the peak type is selected as Gaussian model, and a series of rigorous steps, such as baseline correction, peak searching, automatic peak fitting, etc., are taken in order to achieve accurate peak fitting of diffraction peak spectra. The XRD profiles of four coal samples with different degrees of metamorphism, namely DNH lignite, HS lean coal, HSW gas coal, and XT anthracite, were fitted and analyzed. After fine processing, peak 002 and γ in the combined peaks were successfully obtained, and the corresponding fitting results are presented in Figs. 5–8, respectively. It is worth noting that with the gradual increase of the metamorphic degree of the coal samples, the 002 peak shows a gradual shift to the right. This phenomenon clearly indicates that the metamorphism can significantly affect the microcrystalline structure of coal and promote its gradual development in the direction of graphitization.

Based on Eqs. (1)–(5), the precise quantitative calculation of each microcrystalline morphological structure parameter in coal can be realized, and the detailed results are presented in Table 3. From the data in this table, the evolution of microcrystalline structure parameters, such as d_{002} , L_c , L_a , N_{ave} and g , can be explored in depth for the raw coals with different metamorphic degree and the heat-treated coal samples. These microcrystalline structure parameters are of key significance for understanding the microstructural changes of coals under different degrees of metamorphism and heat treatment conditions.

As shown in Fig. 9, the positions of the 002 diffraction peaks in the X-ray diffraction patterns of the heat-treated coal samples at room temperature and at 700 °C show a significant tendency to shift to a higher angle (2θ value) under a gradually increasing degree of metamorphism. This shift profoundly reflects that the aromatic layer stacking gap, i.e., the crystal plane spacing d_{002} , is continuously shrinking in the microscopic components of the coal. Closely related to this is the fact that the degree of graphitization (g) of coal shows an increasing trend. This change reveals the key role of aromatization in the complex process of coalification. Under the influence of arylation, the intramolecular microcrystalline structure undergoes a significant change, and the arrangement between them becomes more and more compact, which is achieved by significant condensation, showing a tendency of gradual convergence to the graphite crystal structure.

In addition, when we carefully observe and compare the changes in the microcrystalline structure of the coal before and after the heat treatment, we can find a remarkable phenomenon: the degree of crystal surface stacking (L_c), the degree of crystal surface ductility (L_a), and the average number of aromatic layers (N_{ave}) show a tendency to increase at the same time. It clearly demonstrates that the effects produced by coaling are multifaceted. On the one hand, coaling significantly

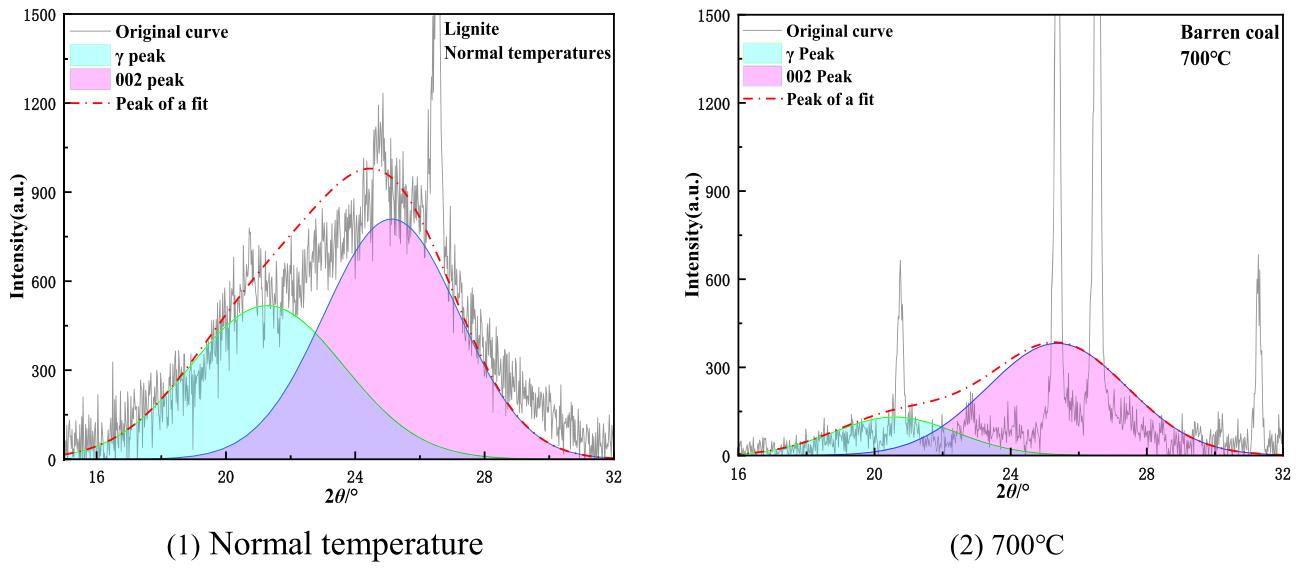


Fig. 5. Lignite split-peak fitting profile.

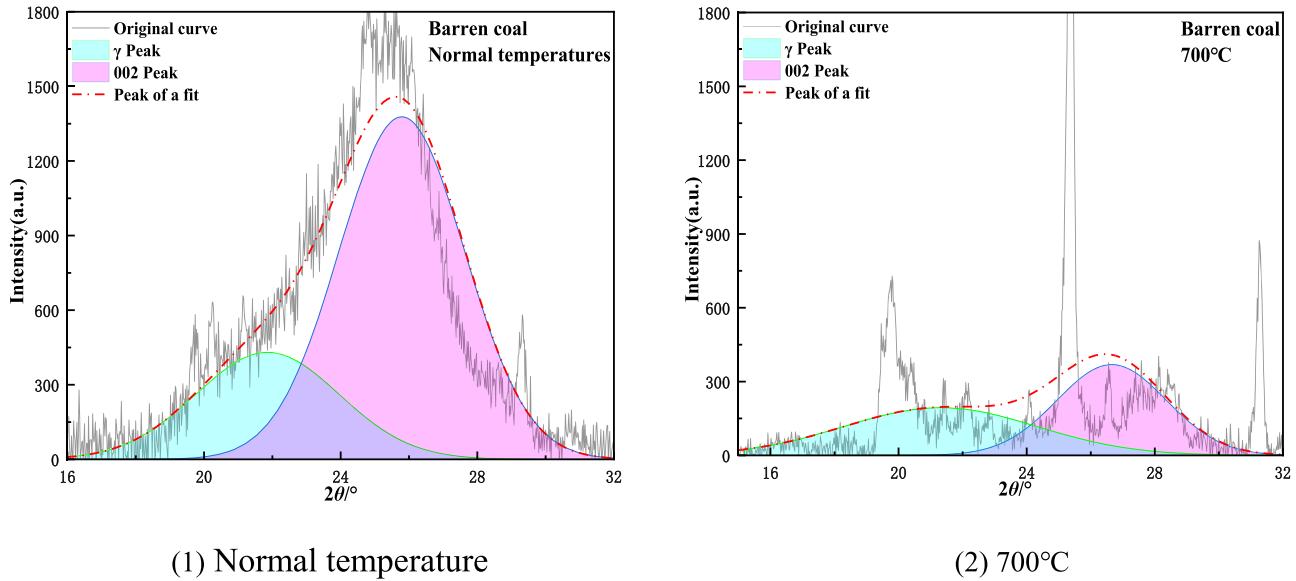


Fig. 6. Mapping of barren coal split peak fits.

enhances the degree of close packing of the microcrystalline molecules, making the interactions between the microcrystalline molecules tighter and more organized. On the other hand, this effect also has a promoting effect on the growth of microcrystalline size. These changes in the microcrystalline structure imply that the high temperature environment plays a key role in the metamorphic process of coal. As a result of the high temperature, the van der Waals forces between the molecules are enhanced, and this enhancement makes the mutual attraction between the molecules increase, which promotes them to come together more closely. At the same time, the chemical bonds are stronger and less likely to break. These two factors work together, which in turn leads to tighter stacking between aromatic layers. The result of this tight stacking suggests that at high temperatures, the coal forms a more ordered carbon layer structure.

3.3. Analysis of the pattern of changes in the microcrystalline structure of coal on its electrical properties

Under room temperature conditions, most of the physical phases in

the coal samples exist in solid form, and when the coal samples are pyrolyzed at a high temperature of 700 °C, they will show a molten state. This state transition is accompanied by significant changes in the microstructure, in which the aromatic layer spacing d_{002} shows a tendency to decrease, while the stacking degree L_c , ductility L_a and the number of aromatic layers N_{ave} increased. These microstructural changes specify that the stacking of carbonaceous layers becomes denser in the presence of high temperatures. This densification leads to an intensification of the van der Waals forces between the layers, which in turn leads to a shortening of the aromatic layer spacing. At the same time, the interlayer electronic coupling is enhanced, and this change elevates the dielectric constant of the coal. In addition to this, this change in microstructural conditions triggered other effects. Both the dielectric loss and the tangent of the dielectric loss increase, which means that the obstacles to the charge transport pathway are also elevated. It is worth noting that the rise in the crystal stacking degree L_c implies that the contact points of the molecules between the carbon layers become more extensive. This extensive contact creates favorable conditions for the formation of electric dipoles, which increases the

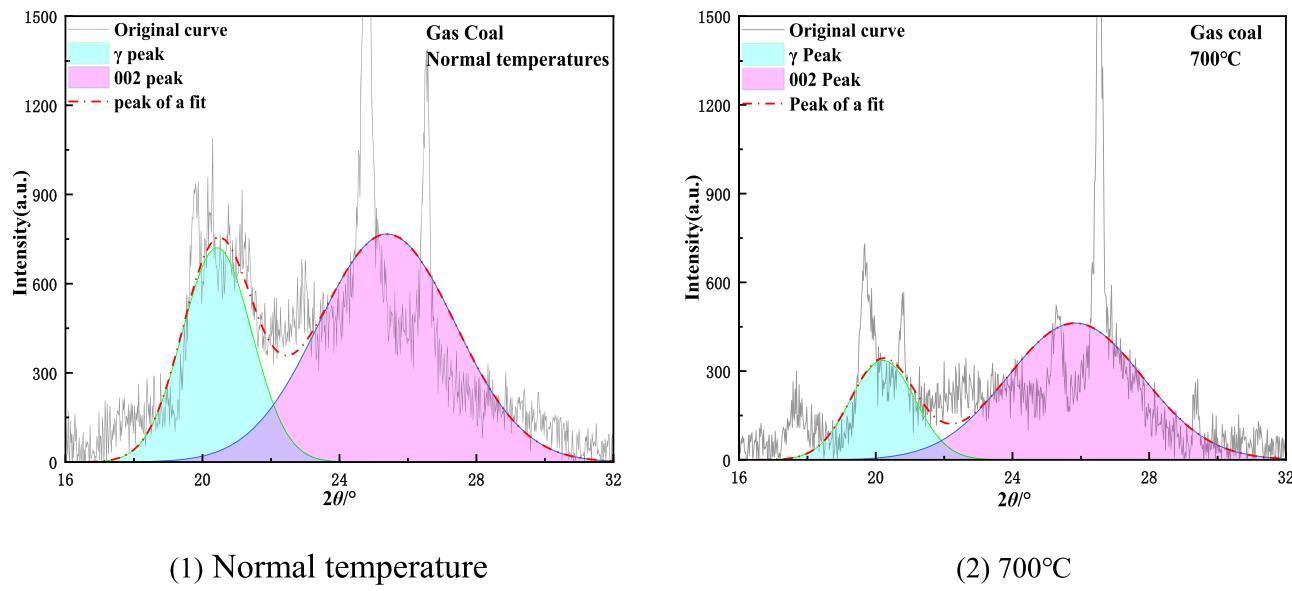


Fig. 7. Gas coal split peak fitting profile.

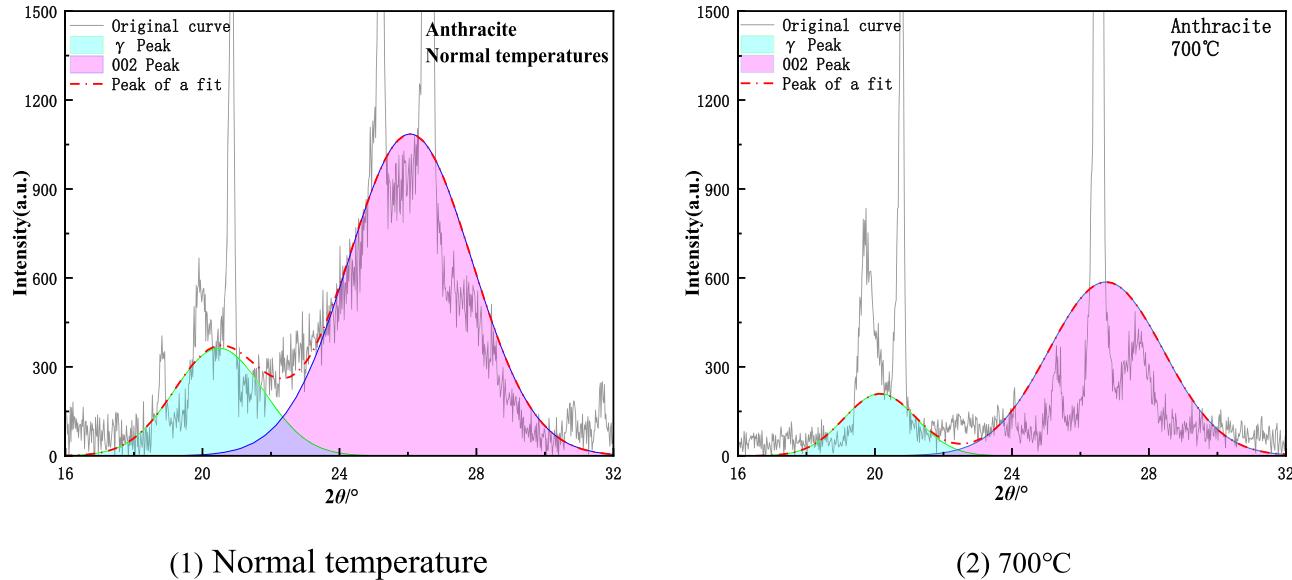


Fig. 8. Anthracite split-peak fitting mapping.

Table 3
Microcrystalline structural parameters of coal.

Coal Samples	Processing Temp	$2\theta_{002}$ / $^{\circ}$	$2\theta_{100}$ / $^{\circ}$	d_{002} /nm	L_c /nm	L_a /nm	N_{ave}	g
Lignite Coal	Normal Temp	25.13	42.54	0.35408	1.63926	1.47577	5.62958	0.69912
	700 °C	25.37	42.87	0.35078	1.66032	1.48624	5.73311	0.75219
Barren Coal	Normal Temp	25.79	43.87	0.34517	1.85697	1.65806	6.37987	0.84266
	700 °C	26.67	44.38	0.33422	2.00331	1.70796	6.99393	1.01894
Gas Coal	Normal Temp	25.39	43.08	0.35051	1.71703	1.52899	5.89857	0.75656
	700 °C	25.83	43.62	0.34464	1.73331	1.55224	6.02927	0.85112
Anthracite	Normal Temp	26.07	44.06	0.34152	1.89736	1.71605	6.55554	0.90134
	700 °C	26.75	44.61	0.33299	2.04950	1.76427	7.15471	1.03869

energy dissipation and leads to the deepening of the polarization. With the increase of the ductility L_a , the interaction between coal molecules is enhanced, which has a positive effect on the polarization phenomenon. The increase of graphitization g usually symbolizes the accumulation of aromatic lamellae and the formation of a more perfect π -electron

conjugation system. This well-established structural change triggers an elevated dielectric constant and also leads to an increase in dielectric loss and dielectric loss tangent. Based on the microcrystalline structure analysis of coal, there is a clear correlation between the microcrystalline structure parameters and the dielectric parameters before and after its

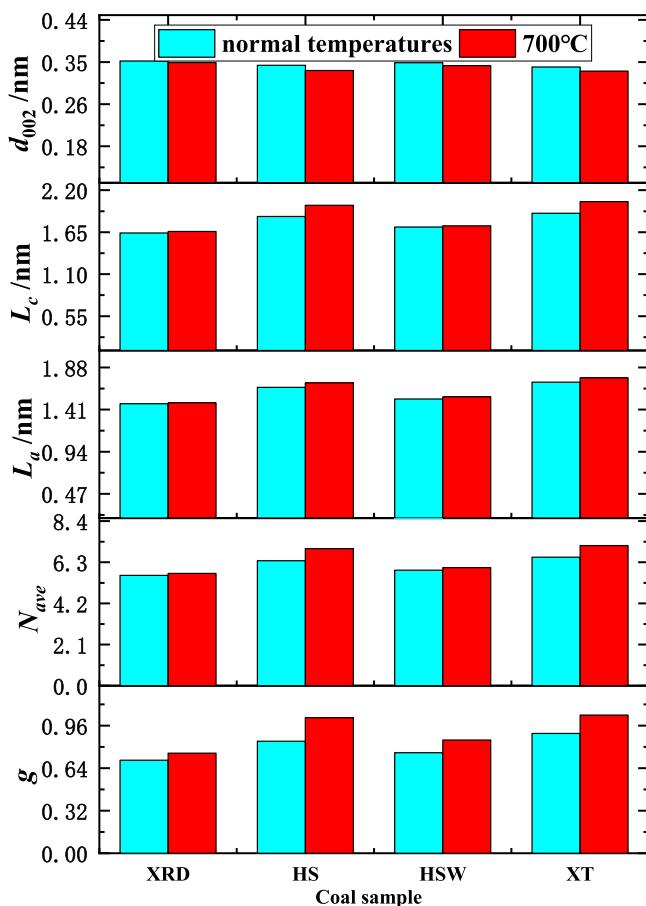


Fig. 9. Microcrystalline structural parameters of heat treated coal samples at room temperature and 700 °C.

heat treatment.

Coal at room temperature has a high electrical resistivity, mainly because it consists mainly of organic matter. Coal at room temperature has a large aromatic layer spacing, d_{002} , as well as relatively low stacking degree, L_c , ductility, L_a , the number of aromatic layers, N_{ave} , and relatively low graphitization, which reflects a looser and more disordered carbon structure. Electrons are scattered and impeded to a greater extent as they pass through this disordered structure, and thus the coal exhibits a higher resistivity. When coal is subjected to high temperatures, the original microcrystalline structure changes. The increase in temperature leads to the release of volatile components in coal and the reorganization of the microcrystalline structure, the aromatic layers of coal will increase, the spacing between the layers decreases, generating larger microcrystalline sizes and forming a more compact and ordered structure, the ordered carbon structure reduces the scattering of electrons and improves the electron mobility, at the same time, the increase of the temperature increases the number of carriers (e.g., electrons and holes) at the same time, which further reduces the electrical resistivity. Overall, the difference in resistivity between coal at ambient and high temperatures directly reflects the close connection between its microcrystalline structure, degree of graphitization and electron conductivity.

4. Physical phase evolution of coal rocks at different temperatures

4.1. Physical studies of coal

In order to investigate the properties of the physical phase

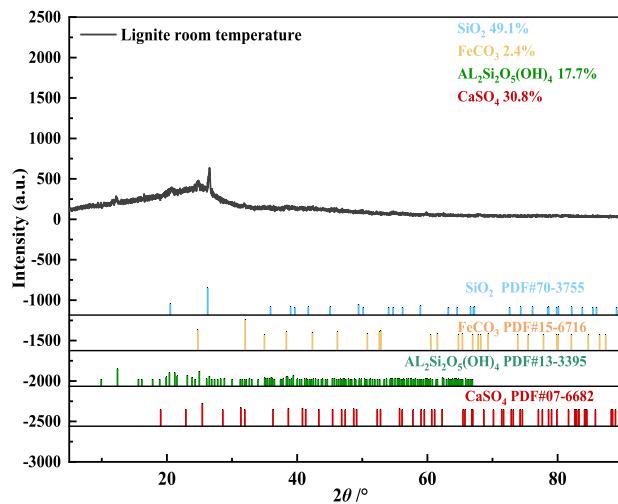
transformation of four coals with different degrees of metamorphism undergoing high-temperature pyrolysis, X-ray diffraction (XRD) was used in this study to collect the X-ray diffraction data of coal samples with different degrees of metamorphism before and after high-temperature heat treatment. The diffraction full spectrum obtained was analyzed in depth using JADE software, and the Rietveld refinement program combined with the PDF database was applied to accurately identify the multiple physical phases present in the coal samples and perform a semi-quantitative evaluation of the composition of the physical phases. Physical phase evolution refers to the transformation of the internal mineral components (e.g., the ferromagnetic compounds Fe_3O_4 , Fe_2O_3 , etc.) of a coal rock body during temperature change as a result of thermal decomposition, redox. This evolution is the core driving force of the magnetization anomalies, and the dynamic changes in the type, content and distribution of internal mineral components (e.g. Fe_3O_4 , Fe_2O_3 , etc.) due to thermal decomposition, redox reaction, or crystal reconstruction during temperature change.

As illustrated in Figs. 10–12 through Fig. 13, following a 700 °C heat treatment, the relative content of lignite quartz, kaolin, and other components in the coal decreased at room temperature. This decrease was accompanied by an increase in the relative content of ferromagnetic compounds, which rose from 2.4 % to 5 %. This included newly generated Fe_2O_3 , which exhibits strong magnetic properties. The relative content of lean coal, which is defined as coal with a low ash and sulfur content, increased from 3 %. At room temperature, the decomposition of rhodochrosite (FeCO_3) occurs, resulting in a physicochemical transformation. The content of ferromagnetic compounds increases to 6.7 %, while the original FeCO_3 decreases to 1.5 %. The relative contents of newly generated magnetite (Fe_3O_4) and hematite (Fe_2O_3) are 2.5 % and 2.7 %, respectively. At room temperature, gas coal exhibited a hematite (Fe_2O_3) content of 0.2 %. Following the heating reaction, this value increased to 10.2 %. The anthracite coal, when observed at room temperature, was found to contain 1.8 % pyrite (FeS_2) and 0.5 % magnetite (Fe_3O_4). Following the application of a heat treatment at 700 °C, the pyrite content was observed to have decreased to 0.4 %, while the relative content of magnetite increased to 0.6 %. Additionally, hematite (Fe_2O_3) was produced with a relative content of 4 %.

4.2. Anomalous law of magnetization rate due to physical phase changes of coal

Subsequent to undergoing high-temperature heat treatment, coal is subjected to a series of complex chemical-physical changes. The relative content and distribution of ferromagnetic compounds within the coal undergoes significant changes, which directly affect the magnetization rate of the coal. During thermal treatment, iron minerals are transformed into more magnetically ferromagnetic compounds, such as hematite (Fe_2O_3) and magnetite (Fe_3O_4), or new ferromagnetic substances are formed by reaction with organic and inorganic components present in the coal. The increase in these ferromagnetic phases and the formation of new ferromagnetic phases enhance the magnetizability of the coal, which in turn results in an overall magnetization rate. The enhanced responsiveness of the coal samples to the external magnetic field can be attributed to two factors. Firstly, the increased ferromagnetic substances imply a greater concentration of iron elements in the magnetic form, which are readily magnetized in the magnetic field. Secondly, the heat treatment alters the size and configuration of the magnetic domains within the coal samples, optimizing their distribution and reducing the internal antimagnetic field, thereby enhancing the macroscopic magnetization effect.

Furthermore, the high-temperature conversion of ferrous minerals to phases with enhanced magnetic properties and the interaction of iron with other components in the coal not only increased the total amount of magnetic substances, but also improved the magnetic properties of the coal by modifying its crystal structure and electronic configuration. Consequently, the alteration in the magnetization rate of coal samples



(1) Normal temperature coal samples

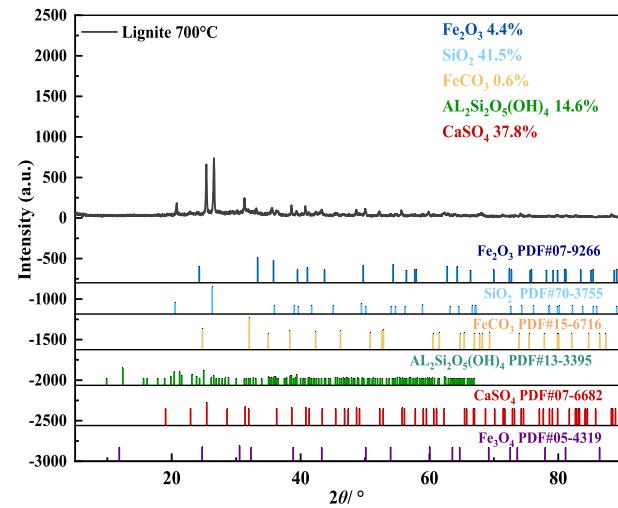
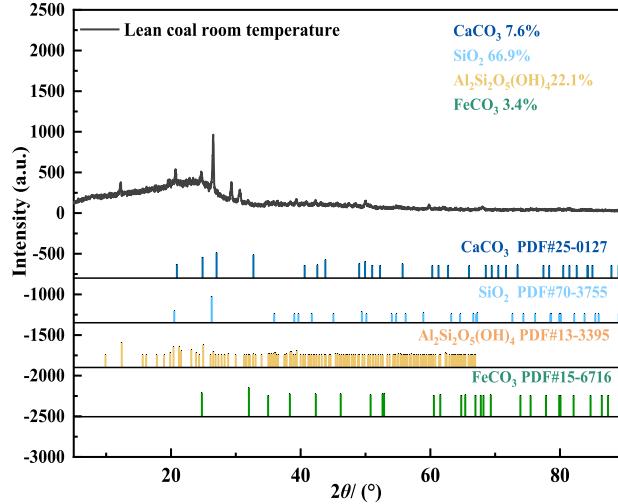


Fig. 10. Lignite physical phase analysis profile.



(1) Normal temperature coal samples

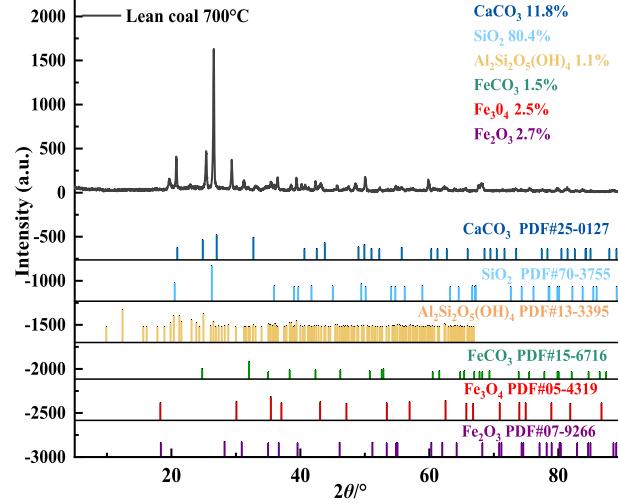


Fig. 11. Lean coal physical phase analysis profile.

subsequent to high-temperature treatment is indicative of both an increase in the quantity of ferromagnetic compounds and an optimization of their internal structure and magnetic characteristics. This provides a composite physicochemical perspective that facilitates a comprehensive understanding of the transformation in coal magnetism.

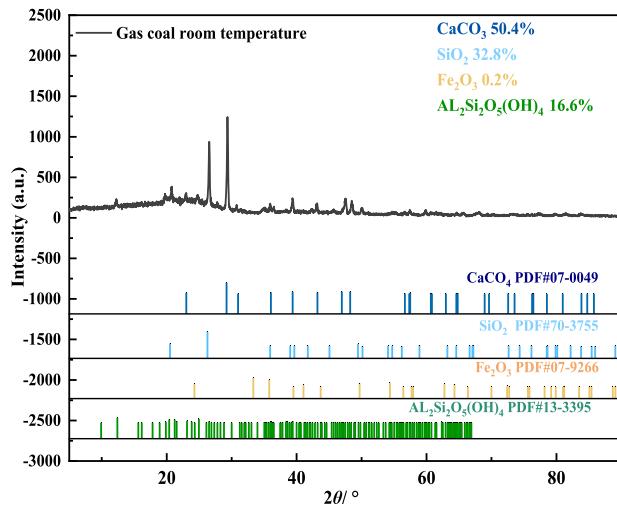
4.3. Petrophysical analysis

In order to clarify the anomalous response mechanism of the change of magnetization in rocks during the warming process, six of the rock samples were selected as the research objects of the magnetic anomaly evolution mechanism, and were scanned and tested *in situ* by X-ray diffractometer during the warming process. The temperature-dependent analysis of the magnetization rate of the rock samples can reveal the change of the magnetic physical phase, and for this reason, four testing temperature points are set according to each rock sample type: room temperature, Tr_{k1} , Tr_{k2} , Tr_{k3} and down to room temperature.

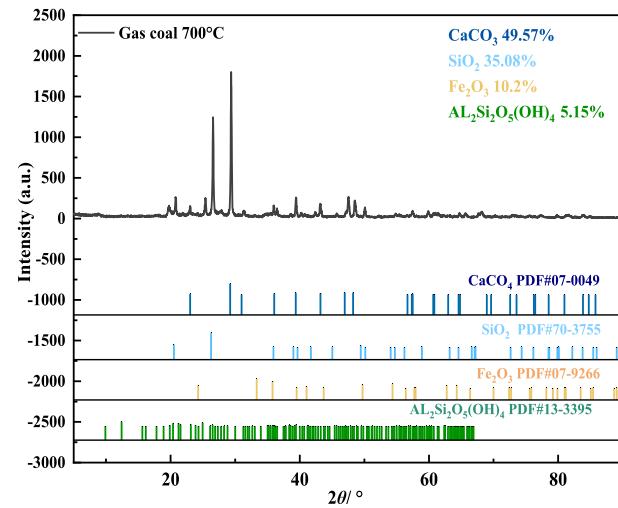
As illustrated in Fig. 14, the relative content of magnetic compounds

in the rock sample R1 at room temperature was 1.8 %, and the ferromagnetic phase exhibited growth to 2.29 when the temperature was increased to 540 °C. As the temperature increased to 700 °C, the relative content of magnetic compounds exhibited a slight decline, reaching 2.05 %. During the subsequent cooling process, the content of ferromagnetic compounds at a temperature drop to 540 °C demonstrated a notable increase, reaching 2.3 % and attaining its maximum value at the temperature point of the test. Upon reducing the temperature to room temperature, the content exhibited a further decline, yet remained higher than the initial room temperature.

As shown in Fig. 15, the ferromagnetic compound of rock sample R2 at room temperature is hematite (Fe_2O_3) with a content ratio of 2.6 %, and when the temperature was increased to 540 °C, hematite disappeared and new ferromagnetic compounds with 1.1 % rhodochrosite (FeCO_3) and 4.42 % pyroxene (Fe_7S_8) were formed, and the total content increased to 5.52 %. At 700 °C, the magnetic phase decreases to 2.72 % with 0.8 % rhodochrosite (FeCO_3) and 1.7 % pyroxene (Fe_7S_8), and produces 0.22 % magnetic pyrite (Fe_6S_{10}). The magnetic phase

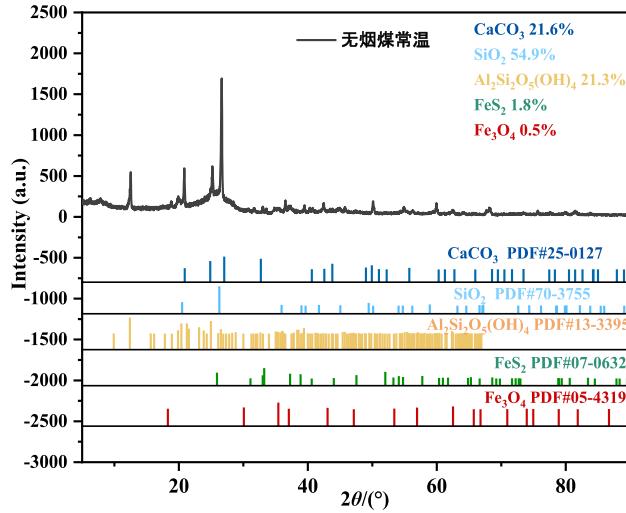


(1) Normal temperature coal samples

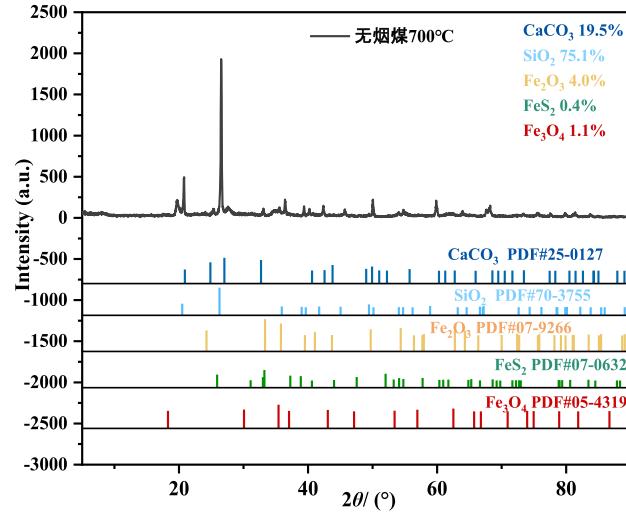


(2) 700°C coal sample

Fig. 12. Gas coal physical phase analysis mapping.



(1) Normal temperature coal samples



(2) 700°C coal sample

Fig. 13. Anthracite physical phase analysis profile.

increased rapidly to 7.51 % when the temperature was lowered to 540 °C. When the temperature returned to ambient, the total magnetic phase was 4.9 %, nearly double the initial ambient temperature, and 2.57 % magnetite (Fe₃O₄) was produced.

As shown in Fig. 16, the ferromagnetic compounds of rock sample R3 are 2.5 % at the initial temperature, containing 1.4 % pyroxene (Fe₇S₈) and 1.1 % hematite (Fe₂O₃), and as the temperature increases to 544 °C, the ferromagnetic phases increase nearly tenfold, with pyroxene increasing to 14.1 % and clinopyroxene (FeCO₃) increasing to 7.7 %. At 700 °C, the total magnetic phase decreases to 2.37 %. As the temperature drops to 540 °C, the magnetic phase rapidly increases to 23.31 %. When the temperature returns to room temperature, the total magnetic phase content is 15.2 %, which is nearly seven times the relative content of magnetic compounds at room temperature.

As shown in Fig. 17, the ferromagnetic compounds of rock sample R5 at the initial temperature had a content ratio of 3.84 %, containing 2.73 % pyroxene (Fe₇S₈) and 1.11 % magnetite (Fe₃O₄). When the temperature was increased to 540 °C, the ferromagnetic phase rapidly

increased to 30.76 %, with pyroxene increasing to 18.27 % and producing 4.8 % rhodochrosite (FeCO₃) and 7.69 % magnetopyrite (Fe₉S₁₀). At a temperature of 700 °C, the total magnetic phase decreases to 1.85 %. When the temperature is lowered to 540 °C, the magnetic phase rapidly increases again to 34.62 %, which is also the maximum value for the percentage of magnetic phase in the measured temperature points. When the temperature returns to room temperature, the total content of magnetic phase is 23.1 %, which is nearly 8 times the relative content of magnetic compounds at room temperature.

As shown in Fig. 18, the ferromagnetic compound of rock sample R7 at the initial temperature is rhodochrosite (FeCO₃) with a content ratio of 0.3 %. When the temperature is increased to 532 °C, the ferromagnetic phase increases to 1.56 %, and the rhodochrosite disappears, producing 0.26 % magnetic pyrite (Fe₉S₁₀), 1.02 % pyroxene (Fe₇S₈), and 0.28 % pyrrhotite. At 700 °C the total magnetic phase decreased to 0.34 %. When the temperature was lowered to 531 °C, the magnetic phase increased again to 1.82 %, containing 0.12 % rhodochrosite and 0.22 % magnetic pyrite, which is also the maximum percentage of

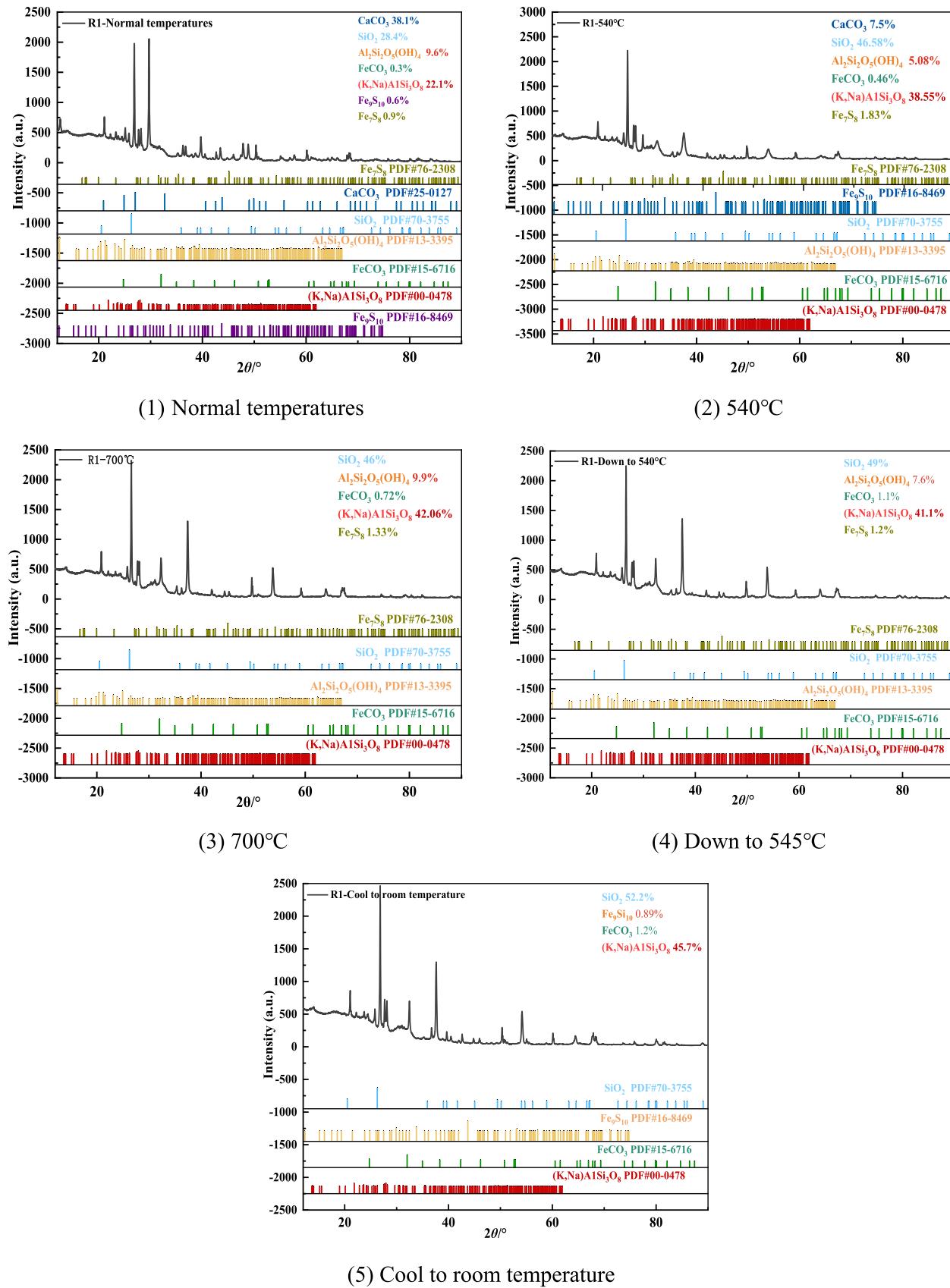


Fig. 14. Plot of R1 physical phase analysis of rock samples during heating process.

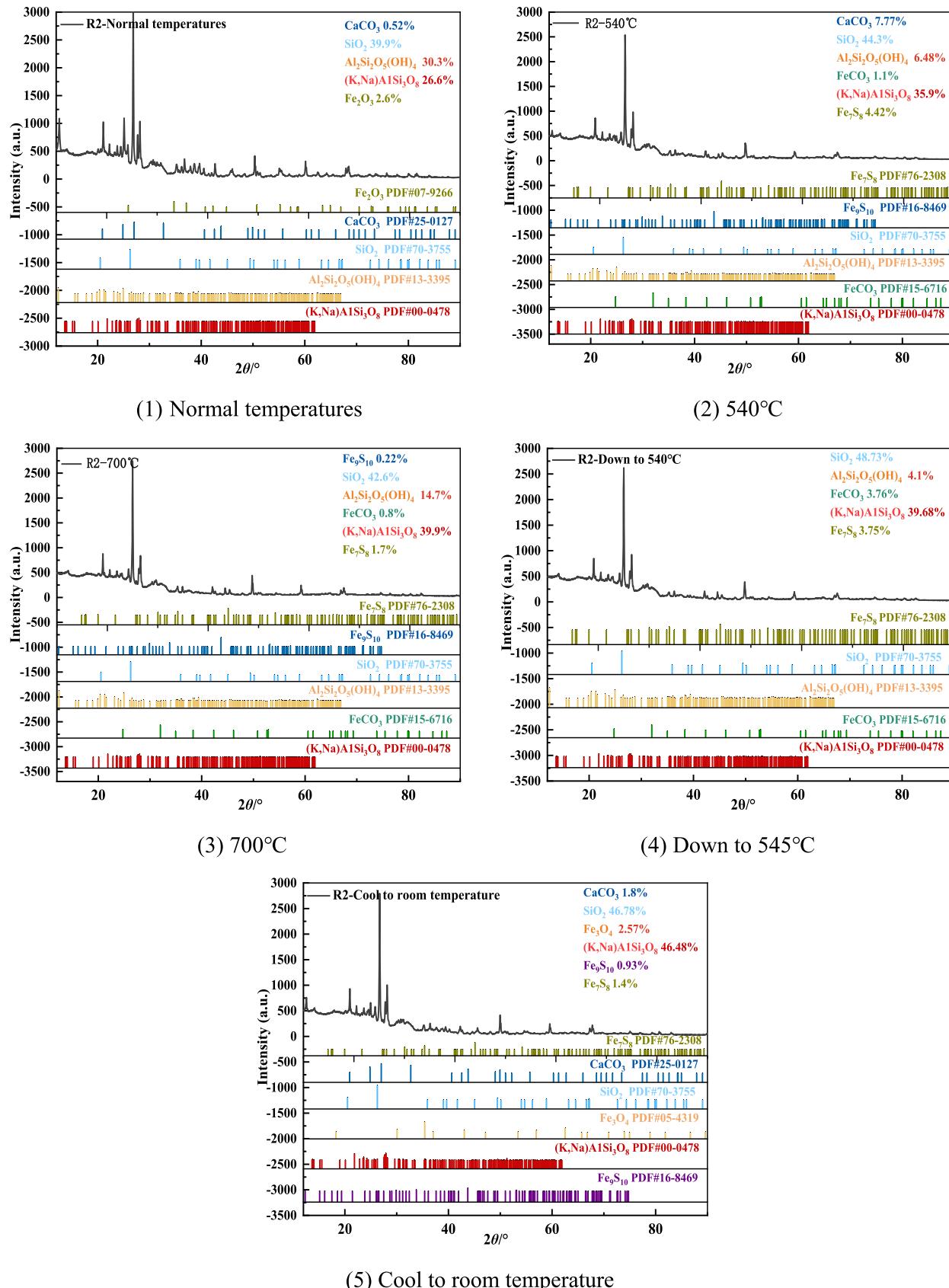


Fig. 15. Plot of R2 physical phase analysis of rock samples during heating process.

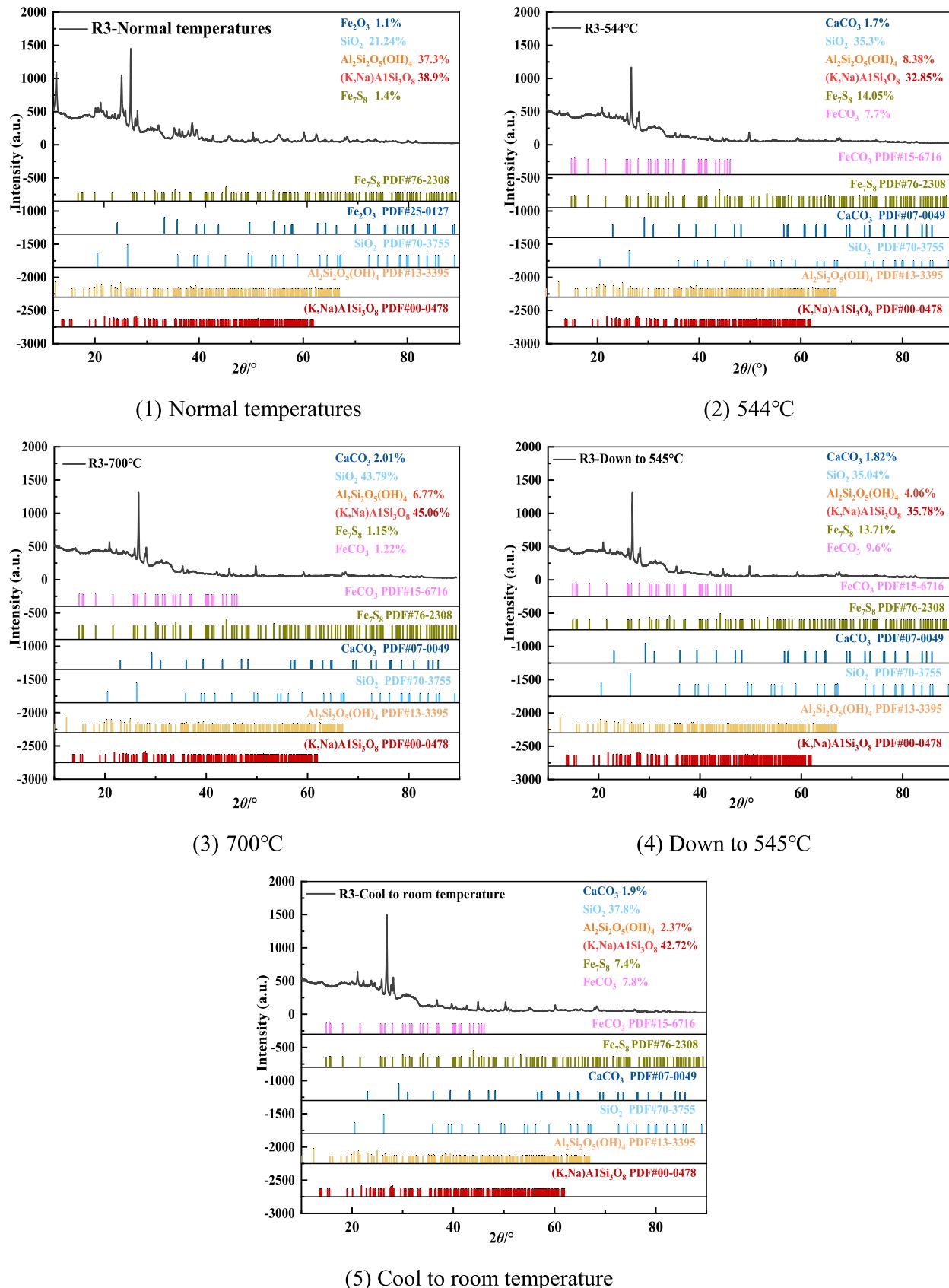


Fig. 16. Plot of R3 physical phase analysis of rock samples during heating process.

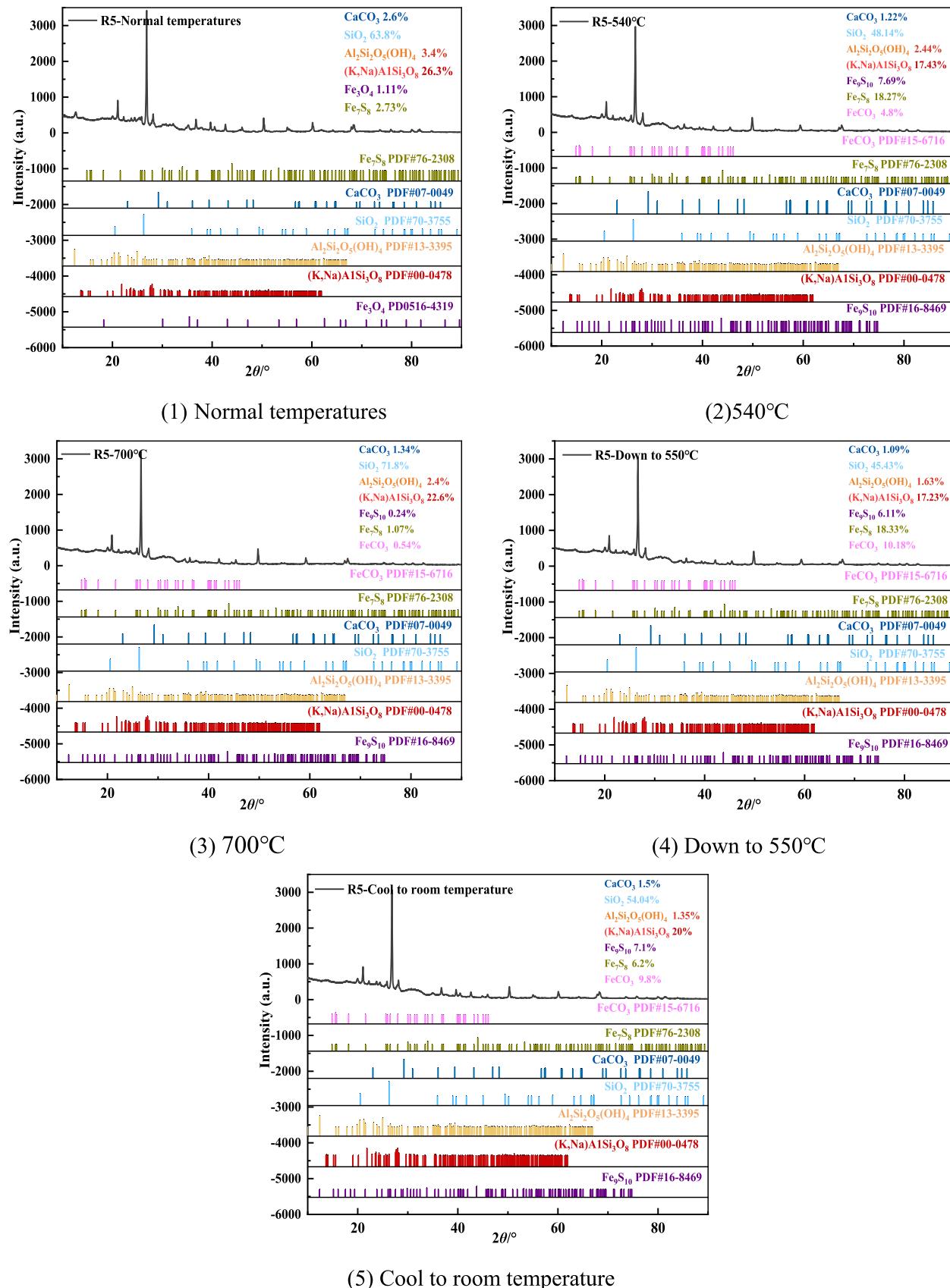


Fig. 17. Plot of R5 physical phase analysis of rock samples during heating process.

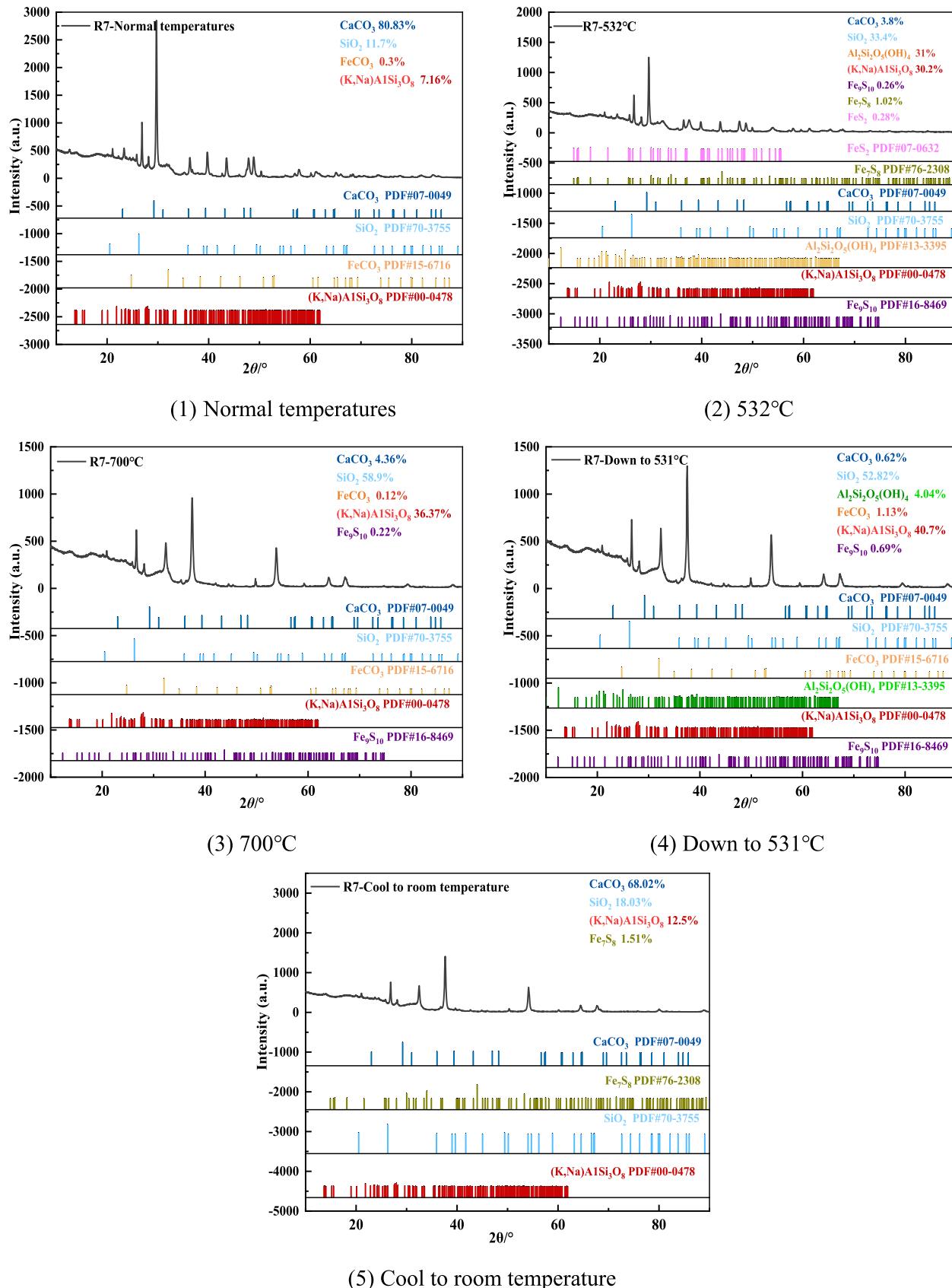


Fig. 18. Plot of R7 physical phase analysis of rock samples during heating process.

magnetic phase among the measured temperature points. When the temperature returns to room temperature, the total magnetic phase content is 1.51 %, at which time there is only one magnetic phase, pyroxene, which is nearly five times the relative content of magnetic compounds at room temperature.

As shown in Fig. 19, the ferromagnetic compounds of rock sample R8 at the initial temperature were 2.3 % magnetopyrite (Fe_9S_{10}) and 1.8 % pyroxene (Fe_7S_8). When the temperature was increased to 540 °C, the ferromagnetic phase increased to 24.65 %, the magnetopyrite-pyroxene disappeared, and the ferrite increased to 11.85 %, producing 12.8 % magnetite (Fe_3O_4). At a temperature of 700 °C, the total magnetic phase decreased to 4.52 %. When the temperature was lowered to 490 °C, the magnetic phase rapidly increased again to 36.88 %, containing 2.07 % rhodochrosite, 5.04 % magnetic pyrite, and 18.4 % pyroxene, which is the maximum percentage of magnetic phase for the temperature points measured. When the temperature returns to room temperature, the total magnetic phase content is 1.51 %, when there are two ferromagnetic phases, 8.84 % pyroxene and the more magnetic magnetite (7.58 %).

As shown in Fig. 20, the ferromagnetic compounds of rock sample R9 at the initial temperature was 1.88 %, containing 0.21 % of rhodochrosite (FeCO_3), 0.52 % of magnetopyrite (Fe_9S_{10}), and 1.15 % of pyroxene (Fe_7S_8) as three magnetic compounds. When the temperature was increased to 495 °C, the ferromagnetic phase increased to 7.55 %, rhodochrosite disappeared, and clinopyrite and magnetopyrite increased to 6.04 % and 1.51 %, respectively. At a temperature of 700 °C, the total magnetic phase decreased to 1.87 %. When the temperature dropped to 480 °C, the magnetic phase rapidly increased to 22.63 %, containing 10.06 % of rhodochrosite, 3.35 % of magnetopyrite, and 9.22 % of pyroxene, which is also the maximum value of the percentage of magnetic phase in the measured temperature points. When the temperature returns to room temperature, the total magnetic phase content is 9.4 %, with 7.2 % rhodochrosite and 2.1 % pyroxene as the two ferromagnetic phases.

The preceding analysis of the relative content of ferromagnetic minerals in the rocks during the change process indicates a close correlation between the temperature change and the transformation or decomposition of the mineral composition. Furthermore, the increase or decrease in the content of ferromagnetic phases directly affects the change in the magnetization rate of the material.

4.4. Anomalous magnetization patterns due to changes in rock physical phases

The change of the rock physical phase on the magnetization rate anomaly during the temperature change process mainly occurs in two major stages: heating and cooling. In the process of rising to $T_{\text{rk}1}$ at room temperature, some ferromagnetic compounds begin to decompose or undergo other chemical reactions. For example, at high temperatures, the relative proportions of the original magnetic compounds increase, and rhodochrosite (FeCO_3) begins to decompose, releasing carbon dioxide and turning into hematite (Fe_2O_3), while pyrite may further undergo chemical reactions to form magnetite. These changes result in an increase in the magnetization of the rock due to an increase in the relative amount of magnetic compounds and the fact that hematite and magnetite are more magnetic than the original ferromagnetic compounds. As the temperature continues to increase toward $T_{\text{rk}2}$, some of the magnetic compounds may become unstable, while at higher temperatures the inert gases are no longer able to inhibit the redox reactions, phase decomposition, and reactions between the different minerals, resulting in the decomposition of the original magnetic compounds or their transformation into non-magnetic or weakly magnetic phases. For example, magnetic pyrite (Fe_9S_{10}) and gray iron ore (Fe_7S_8) may decompose or transform into other phases as a result of elevated temperatures, e.g., pyrrhotite (FeS_2) may further oxidize and decompose into non-magnetic sulfides and iron oxides. These processes directly reduce the amount of the original highly magnetic compounds. This

leads to a decrease in the magnetization of the rock. These phase changes at high temperatures are not only a direct result of decomposition and oxidation of compounds, but may also involve rearrangement and reorganization of the crystal structure, which in turn affects the magnetic properties of the mineral.

During the subsequent cooling process, particularly from $T_{\text{rk}2}$ to $T_{\text{rk}3}$, some of the magnetic phases observed at elevated temperatures undergo decomposition or transformation, resulting in the formation of non-magnetic or less magnetic phases. However, as the temperature decreases, the kinetic conditions for the interconversion of compounds are altered, such that the decomposition products of these minerals may re-react to form more magnetic compounds. For instance, hematite (Fe_2O_3) may undergo a reaction with reducing agents present in the surrounding environment during the cooling process, resulting in the regeneration of a more magnetic phase, such as magnetite (Fe_3O_4), which exhibits a higher magnetization relative to the former. Furthermore, some nonmagnetic or weakly magnetic intermediates may undergo transformation into compounds with enhanced magnetic properties during the cooling process, thereby contributing to an overall increase in the proportion of ferromagnetic compounds. Furthermore, a reduction in temperature results in lattice contraction of magnetic minerals within the rock, which may subsequently lead to remodeling within the lattice. This lattice reconstruction not only optimizes the crystal structure of the minerals but also facilitates the rearrangement and merging of the magnetic domains, thereby enhancing the responsiveness of the magnetic domains to external magnetic fields. The enlargement of magnetic domains and the reduction in the number of magnetic domain walls diminish domain wall scattering within the magnetic material. Furthermore, a reduction in temperature facilitates the alignment of electron spins, thereby enhancing the ferromagnetism of the material. During the cooling process, the spins of unpaired electrons in the minerals tend to align, resulting in enhanced magnetism. This is due to the fact that the temperature reduction reduces the interference of thermal vibrations on the electron spin alignment, thus increasing the overall magnetization. As the temperature of the rock sample decreases to room temperature, magnetic minerals may undergo repair of lattice distortions and reduct.

A comparison of the magnetic phases of the rocks before and after heating revealed that the combination of enhanced magnetic properties of the magnetic minerals resulting from structural reorganization and domain optimization, the stabilization of the chemical composition and the presence of magnetic phases, and the residual magnetization effect led to a higher magnetization rate and content of magnetic compounds in these rocks than in ambient rocks that had not undergone high-temperature treatment.

The series of phase and magnetic changes observed reflects the significant effect of temperature on the behavior of ferromagnetic compounds in rocks. From a chemical perspective, the decomposition and oxidation reactions that occur during the process of warming are the primary drivers of these changes. These reactions directly alter the type and content of ferromagnetic compounds present in rocks. Furthermore, alterations in temperature may also result in modifications to the crystal structure of minerals. These microscopic alterations in structure impact the magnetic domain structure of magnetic minerals, specifically the dimensions and distribution of regions exhibiting uniform magnetic orientation. The optimization of the magnetic domain structure results in a reduction of magnetic domain walls within the material, which in turn leads to a decrease in the antimagnetic field and an enhancement of the macroscopic magnetization of the rock. Moreover, alterations in the crystal structure may influence the arrangement of electrons, which in turn affects the magnetic properties of the material. These changes affect not only the relative content of ferromagnetic compounds, but also their chemical state and crystal structure, thus exerting a profound effect on the overall magnetic properties of the rock. These phase transitions in magnetic minerals are not only reflected in the direct transformation of compounds, but also involve more complex chemical reaction pathways.

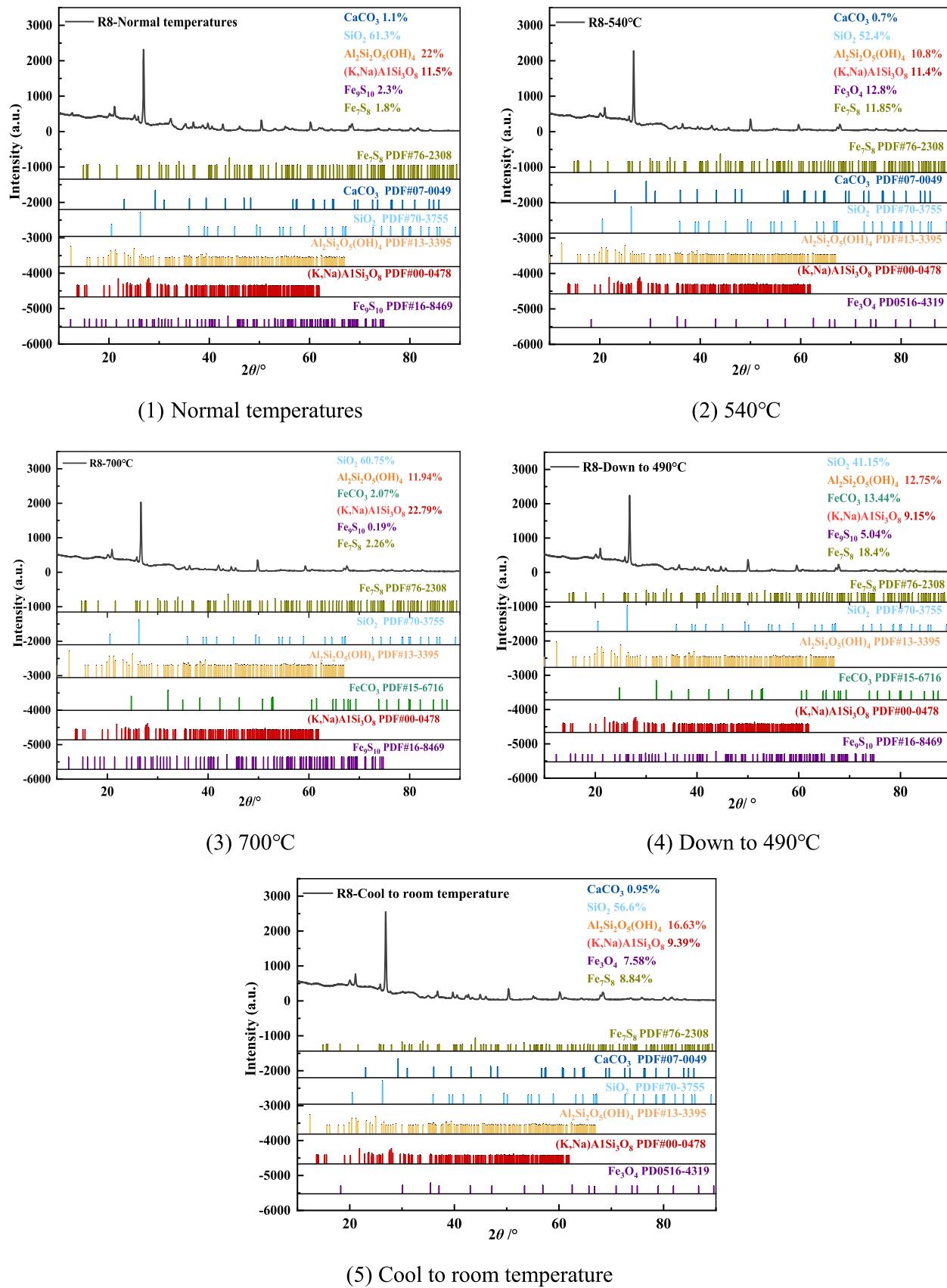


Fig. 19. Plot of R8 physical phase analysis of rock samples during heating process.

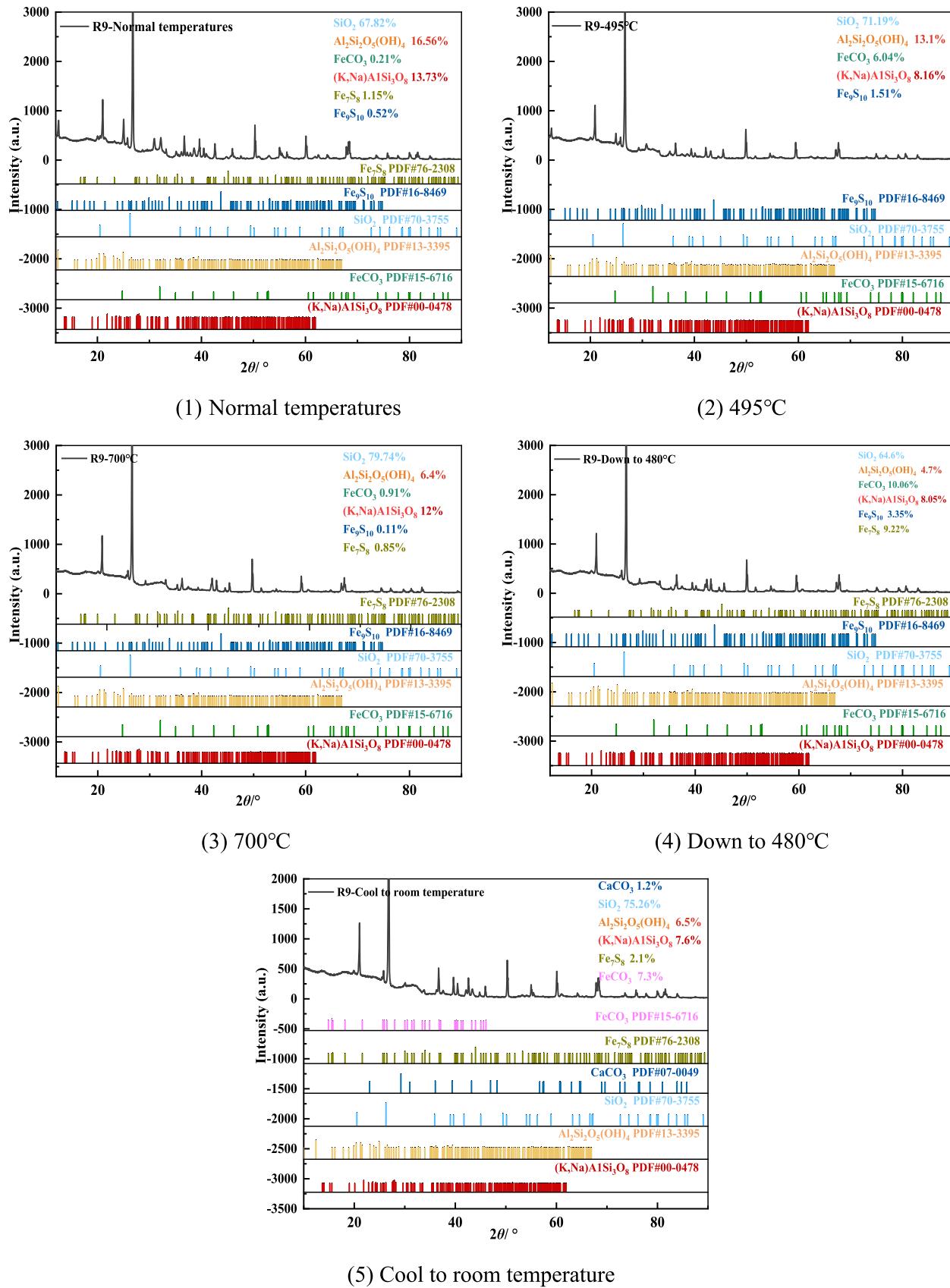


Fig. 20. Plot of R9 physical phase analysis of rock samples during heating process.

For example, the reduction of magnetite in sulfur-bearing environments may produce pyroxene or magnetic pyrite. These reactions and phase transformation processes illustrate the mechanisms through which temperature affects the magnetic properties of rocks, elucidating the behavioral patterns of ferromagnetic compounds under disparate temperature conditions and their contribution to the magnetization of rocks.

In general, the magnetic alterations observed in rocks during warming can be attributed to a complex, multifactorial, and interactive process. This process encompasses the decomposition, oxidation, and reduction of compounds, as well as alterations in the structure of crystals and magnetic domains. These changes reflect the thermal stability and chemical reactivity of ferromagnetic compounds in rocks under different temperature conditions, as well as the manner in which these physicochemical changes interact to influence the magnetizability properties of rocks.

5. Conclusion

- (1) During the pyrolysis process, the evaporation of water at room temperature causes a rapid increase in the resistivity of low-rank coal. The thermal movement of coal molecules is weak, and the dielectric parameters exhibit a weak trend of change with temperature. As the pyrolysis temperature increases, the thermal movement between molecules intensifies, leading to the breaking of chemical bonds and the release of volatiles. This results in an enhancement of the electron migration rate within the coal and a rapid decrease in its resistivity. The establishment of dipole steering polarization is rapid under high temperatures, and the dielectric response of coal is pronounced. Once the pyrolysis reaction is largely complete, the thermal motion of coal molecules declines, reducing the capacity to retain charge. This results in a gradual decline in resistivity and dielectric constant, accompanied by a rapid increase in dielectric loss and dielectric loss tangent.
- (2) At room temperature, the microcrystalline structure of coal tends to become increasingly ordered with the increase of the degree of metamorphism. This, in conjunction with the moisture content of coal, exhibits the characteristics of the electrical anomaly response of coal. Following high-temperature treatment, alterations to the crystal structure of coal are observed, accompanied by a reduction in the spacing of aromatic layers and an intensification of order within the aromatic structure. This phenomenon gives rise to a considerable acceleration in electron migration, which in turn results in a notable decline in the resistivity of coal and an augmentation of its dielectric constant.
- (3) The characteristics of change exhibited by ferromagnetic compounds in coal rock vary depending on the temperature stage. The primary factor influencing this series of phase changes and magnetic changes is the decomposition and oxidation reaction occurring during the warming process. This leads to a change in the type of ferromagnetic compounds present in the coal rock, a change in the content of the original magnetic compounds, and, at the same time, temperature changes result in adjustments to the microstructure, which in turn affect the magnetic domain structure of the magnetic phase. This enhances the magnetization effect of the coal rock. At elevated temperatures, the magnetic compounds become increasingly unstable, undergoing decomposition into non-magnetic or weakly magnetic phases. This results in a rapid decline in the magnetization rate. The kinetic conditions for the interconversion of magnetic compounds undergo a transformation during the process of cooling, which in turn prompts these minerals to undergo further reactions and form additional magnetic.
- (4) The combination of structural reorganization and magnetic domain optimization in the coal rock has resulted in the improvement of magnetic properties of magnetic minerals, the

stabilization of chemical composition, and the enhancement of the stable existence of magnetic phases. Additionally, the residual magnetization effect has led to a higher content of magnetic compounds and a higher rate of magnetization than that of the coal rock in its initial state.

Institutional review board statement

Not applicable.

Informed consent statement

Not applicable.

CRediT authorship contribution statement

Xiaokun Zhao: Writing – review & editing, Software. **Jun Ge:** Writing – original draft, Resources, Investigation, Data curation. **An Zhang:** Validation. **WenCai Wang:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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