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Mineralogy characteristics, stability conditions, and formation pathways of synthetic pyrrhotite formed by heating pyrite at 700°C

Highlights

Synthetic pyrrhotite differs from natural pyrrhotite in the mineral association.
Synthetic pyrrhotite-4C was stable under 0.5–2 h of heating at 700°C in air.
Synthetic pyrrhotite-4C had the largest content at 700°C by heating for 1 hour.
In air, synthetic pyrrhotite-4C is formed mainly via two pathways.
Pyrite → pyrrhotite-4C is the more favorable path of pyrrhotite-4C formation.

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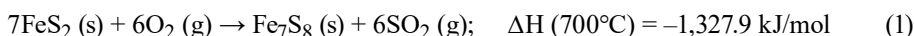
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Introduction

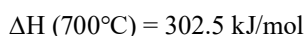
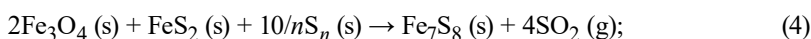
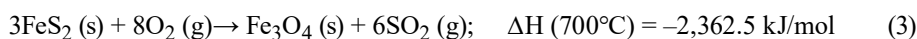
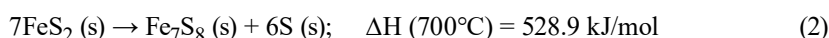
Pyrite (FeS_2) is the most common sulfide mineral and is widely distributed in nature (Priestley and Paul 1964; Craig et al. 1998; Kołodziejczyk 2009; Oliveira et al. 2016; Stepanov et al. 2021). As a result of its universality and importance, pyrite has been extensively studied in terms of its mineralogy, thermal decomposition, and mineral transformation (Lambert et al. 1998; Huang and Rowson 2001; Ferrow and Sjöberg 2005; Wang et al. 2014; Huang et al. 2021). When heated at high temperatures, pyrite may transform into ferrimagnetic minerals (e.g. pyrrhotite or magnetite) or canted antiferromagnetic minerals (e.g. hematite) (Li and Zhang 2005; Wang et al. 2008; Bhargava et al. 2009; Shi et al. 2015; Zhang et al. 2019). Understanding these transformation mechanisms can inform important technical approaches for the magnetic separation of pyrite as a paramagnetic mineral.

Pyrrhotite is widely identified in endogenetic deposits and has also been observed in a series of hydrothermal deposits (Matsumoto and Nakamura 2012; Bogdanova et al. 2016; Palyanova et al. 2019; Mansur et al. 2021). Pyrrhotite, like pyrite, is a raw material used in the preparation of sulfuric acid and sulfur. The mineralogical characteristics of natural pyrrhotite have been extensively studied (Kondoro and Kiwanga 1997; Kontny et al. 2000; Wang and Salveson 2005; Selivanov et al. 2008). However, synthetic pyrrhotite, which is formed by heating pyrite in air, has not been sufficiently investigated. It is necessary to study the crystal structure and mineral association differences between synthetic and natural pyrrhotite, as well as the stability conditions of synthetic pyrrhotite at elevated temperatures in air, based on existing research. These investigations will support processing engineering techniques and industrial applications of pyrite and pyrrhotite.

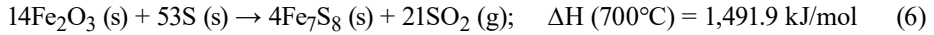
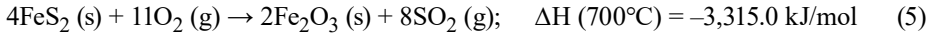
Synthetic pyrrhotite is mainly formed from the oxidation of pyrite at elevated temperatures in air (Wang et al. 2008; Wang et al. 2014). The transformation of pyrite to pyrrhotite is carried out according to the following reaction:



The enthalpy value was calculated by HSC Chemistry 6.0 software. Some possible formation pathways have been proposed by previous studies. For example, small amounts of magnetite may participate in the transformation of pyrite to pyrrhotite (Li and Zhang 2005; Wang et al. 2008):



Small amounts of magnetite cannot be identified by X-ray diffraction (XRD) but can be verified by thermomagnetic curve analysis. According to Wang et al. (Wang et al. 2014), hematite may be further transformed into pyrrhotite in the presence of sulfur produced in reaction (2):



Possible formation pathway should be verified by subsequent experiments.

In this work, natural pyrite, pyrrhotite, and hematite samples were used for analysis. The synthesis pathway of synthetic pyrrhotite was verified by material synthesis. The crystal structure, mineral association, and formation pathways of pyrrhotite formed by heating pyrite were further examined using XRD.

1. Experimental method

Information on the experimental samples is presented in Table 1. Table 2 provides chemical compositions of natural mineral samples as analyzed through the use of X-ray fluores-

Table 1. Natural mineral samples and reagents from China used in the experiments

Tabela 1. Naturalne próbki minerałów i odczynniki z Chin użyte w doświadczeniach

Minerals	Samples	Chemical formula	Sample characteristics	Location
Pyrrhotite	KW28	Fe_{1-x}S	single mineral aggregate	Yuhang, Zhejiang
Pyrite	KW30	FeS_2	single mineral aggregate	Kuandian, Liaoning
Kidney-shaped hematite	KW51	Fe_2O_3	single mineral aggregate	Xuanhua, Hebei
Sulfur		S	AR	Chengdu, Sichuan

Table 2. Chemical compositions (wt.%) of natural mineral samples

Tabela 2. Składy chemiczne (% wag.) naturalnych próbek mineralnych

Minerals	Samples	Fe_2O_3	SO_3	SiO_2	Al_2O_3	CaO	MgO
Pyrrhotite	KW28	32.58	66.38	0.72	0.13	0.19	0
Pyrite	KW30	34.39	64.62	0.74	0.17	0.05	0.03
Kidney-shaped hematite	KW51	76.78	0.03	21.49	1.15	0.32	0.24

cence (XRF). Natural pyrite (KW30) had a pure pyrite phase (Figure 1). As Wang et al. (Wang et al. 2014) reported the highest conversion efficiency from pyrite to pyrrhotite in air at 700°C, sample KW30 was crushed to 40 mesh and heated at 700°C for 0.5–4 h in air prior to further experiments. One sample was weighted for 5 g into a corundum crucible and heated using a muffle furnace. K-type thermocouple was utilized to measure the temperature with a deviation of $\pm 5^\circ\text{C}$.

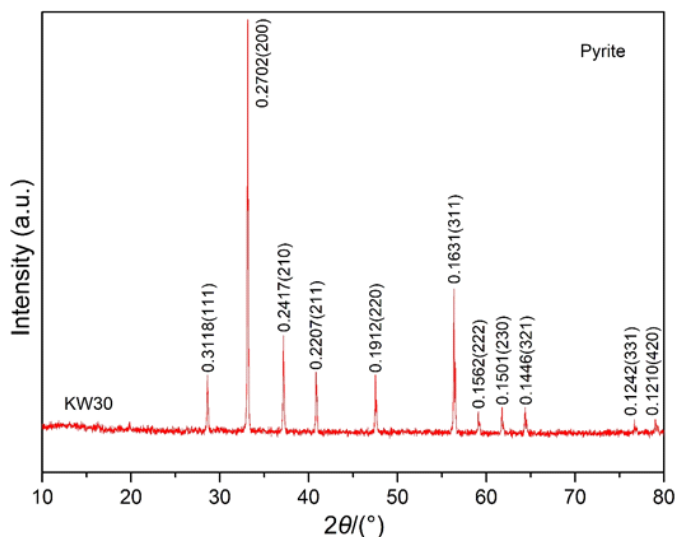


Fig. 1. XRD pattern of natural pyrite (KW30) (modified from Wang et al. 2014)

Rys. 1. Dyfraktogramy XRD naturalnego piryty (KW30)

Solid-phase reaction experiments for reaction (6) were conducted as follows: natural hematite (KW51) and sulfur were crushed and ground to a 200 mesh size using an agate mortar. They were mixed evenly in a molar ratio of 1:3.8. The mixture was heated at 700°C for 1 h in air.

Powder XRD experiments were performed on a DX-2700 diffractometer (Haoyuan Instrument Co., Ltd., China) to determine the constituent phases of the samples. The diffraction profiles were recorded in the range of 10–80° (2θ) using a step scan technique (1 s per step with an interval of 0.03°) under Cu K_α radiation. The XRD results were analyzed using MDI Jade 6 software with a diffraction standards database (ICDD PDF2 2004).

2. Results and discussion

2.1. Mineralogy characteristics of natural and synthetic pyrrhotite samples

FeS is troilite and is hexagonal; Fe₇S₈ is one of the typical structures of pyrrhotite mineral and is monoclinic pyrrhotite-4C. Pyrrhotites with intermediate compositions (5C, 6C, and their intermediates) are monoclinic but mentioned as pseudo-hexagonal (Morimoto et al. 1975). As shown in Figure 2, the natural pyrrhotite sample (KW28) has pyrrhotite-4C and magnetite phases as well as unknown phases, causing some unidentified diffraction peaks at 10–13° and 27–29° (2θ). Pyrite products heated at 700°C for 1 h consisted of mainly pyrrhotite-4C and hematite with a small amount of pyrite and magnetite as well as unknown phases inducing diffraction peaks at 25–27° (2θ). There was a difference in the paragenetic association of minerals between natural and synthetic pyrrhotites. The XRD results matched well with those of JCPDS 89-1954, pyrrhotite-4C (Tokonami et al. 1972). Diffraction peaks of (228), (400), (224), (620), (448), and (004) were clearly observed. The *d*-values for the hexagonal form (102), distinct from the monoclinic form (228) of pyrrhotite-4C at 44° (2θ) in this case, were 0.2056 nm for natural pyrrhotite-4C and 0.2054 nm for synthetic pyrrhotite-4C (Figure 2). The values obtained are in good agreement with that of 0.2052 nm obtained from a monoclinic pyrrhotite-4C crystal (Tokonami et al. 1972). This indicates

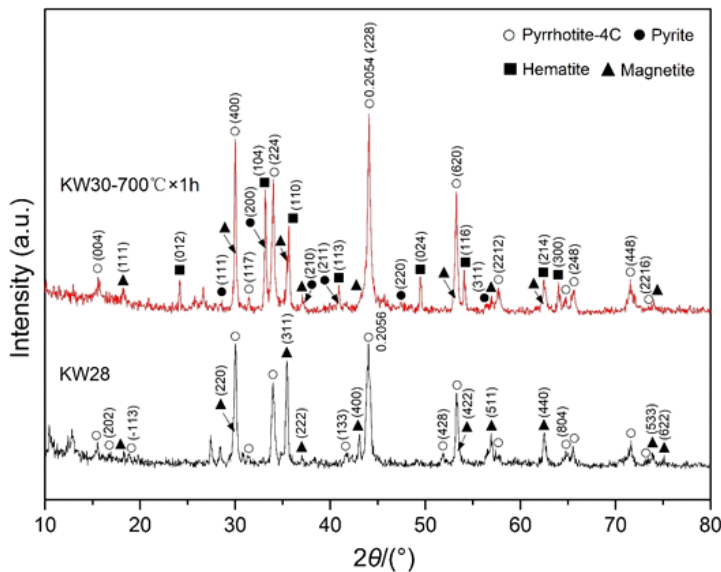


Fig. 2. XRD patterns of natural pyrrhotite (KW28) and synthetic pyrrhotite formed by heating pyrite (KW30) for 1 h at 700°C

Rys. 2. Dyfraktogramy XRD pirytynu naturalnego (KW28) i pirytynu syntetycznego powstałe w wyniku ogrzewania pirytu (KW30) przez 1 h w temperaturze 700°C

that the natural and synthetic pyrrhotites may both be classified as pure pyrrhotite-4C. Table 3 lists the lattice parameters of natural and synthetic pyrrhotite-4C. All samples were monoclinic pyrrhotite-4C (Fe_7S_8) and exhibited minimal differences in terms of lattice parameters.

Table 3. Lattice parameters of natural pyrrhotite (KW28) and synthetic pyrrhotite formed by heating pyrite (KW30) for 1 h at 700°C

Tabela 3. Parametry sieci krystalicznej pirotytynu naturalnego (KW28) i pirotytynu syntetycznego utworzonego przez ogrzewanie piryty (KW30) przez 1 h w temperaturze 700°C

Samples	Crystal system	$a \times b \times c$ (nm)	β	V (nm^3)	Z	D
Natural pyrrhotite	Monoclinic	$1.1910(7) \times 0.6863(4) \times 2.2825(13)$	$90.43(3)^\circ$	1.866	8	4.610
Synthetic pyrrhotite	Monoclinic	$1.1912(10) \times 0.6860(5) \times 2.2774(19)$	$90.44(5)^\circ$	1.861	8	4.621

2.2. Stability conditions of synthetic pyrrhotite-4C formed under 700°C in air

According to previous studies, synthetic pyrrhotite formed by heating pyrite at 700°C is stable under argon and nitrogen atmospheres (Li and Zhang 2005; Wang et al. 2008; Bhargava et al. 2009; Shi et al. 2015). However, it is unclear whether synthetic pyrrhotite remains stable in air. At 700°C, synthetic pyrrhotite formed by heating pyrite had the maximum content in air (Wang et al. 2014).

As shown in Figure 3, the pyrite-heated products have the phases of mainly pyrrhotite-4C and hematite when held at 700 °C in air. Synthetic pyrrhotite-4C formed stably by heating pyrite for 0.5–2 h. Its relative content was the highest at 81.8% (Table 4) by heating for 1 h (Figure 4). The percentages of pyrrhotite-4C and hematite were calculated by MDI Jade 6 software. Small amounts of pyrite and magnetite were also observed. However,

Table 4. The percentages (wt.%) of pyrrhotite-4C and hematite in pyrite products by heating at 700°C

Tabela 4. Udziały procentowe (% wag.) pirotytynu-4C i hematytu w produktach pirotytowych po ogrzaniu w temperaturze 700°C

Heating time	0.5 h	1 h	2 h	3 h	4 h
Pyrrhotite-4C	47.8	81.8	44.2	0	0
Hematite	52.2	18.2	55.8	100	100

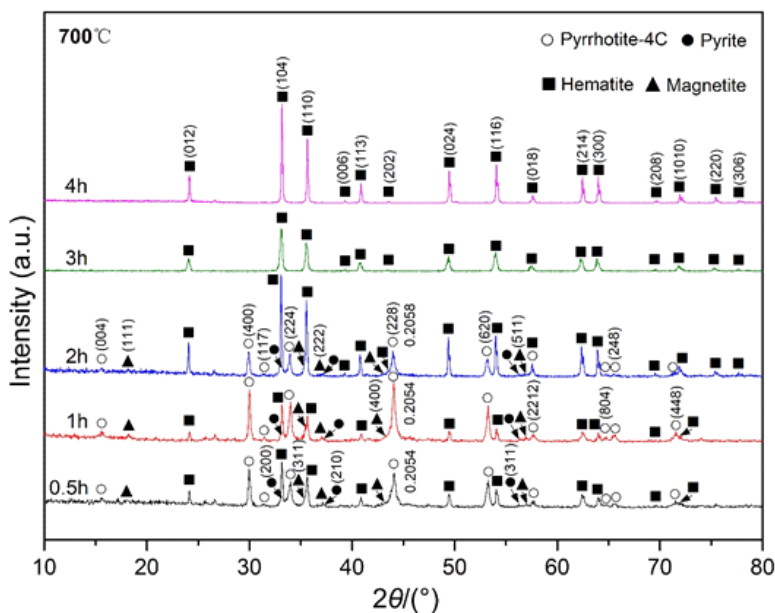


Fig. 3. XRD patterns of pyrite products formed by heating at 700°C for 0.5 to 4 h

Rys. 3. Dyfraktogramy XRD produktów pirytowych powstałych w wyniku ogrzewania w temperaturze 700°C w czasie od 0,5 do 4 godzin

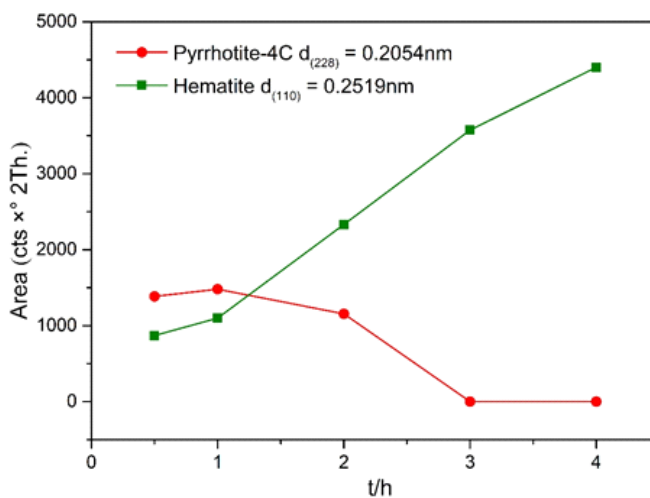


Fig. 4. Integral areas of diffraction peaks of pyrrhotite-4C at $d_{(228)} = 0.2054$ nm and hematite at $d_{(110)} = 0.2519$ nm in pyrite products by heating at 700°C for 0.5 to 4 h

Rys. 4. Obszary integralne pików dyfrakcyjnych pirytynu-4C przy $d_{(228)} = 0,2054$ nm i hematytu przy $d_{(110)} = 0,2519$ nm w produktach pirytowych po ogrzewaniu w temperaturze 700°C w czasie 0,5 do 4 h

the pyrrhotite-4C phase disappeared completely when the heating period exceeded 3 h, as was the case for pyrite and magnetite. Hematite was the final oxidation product, which is consistent with several previous studies (Wang et al. 2008; Bhargava et al. 2009). Li and Zhang (Li and Zhang 2005) suggested that pyrrhotite is the final product in air. This difference may be due to the creation of an anoxic environment by the slender quartz glass tubes (with an inner diameter of 6.5 mm and length of 177 mm) used in the heating experiments.

It should be noted that the pyrite heating process in this work was always performed in a muffle furnace. Using other type of furnaces, e.g. fluidized bed, microwave furnace, may have different stability conditions for synthetic pyrrhotite-4C in air. This is worthy of further study in future work.

2.3. Formation pathways of synthetic pyrrhotite-4C formed by heating pyrite at 700°C in air

In this study, synthetic pyrrhotite-4C was mainly formed from the oxidation of pyrite in air by reaction (1). There are several possible formation pathways. As illustrated in Figures 2 and 3, the XRD results show that a small amount of magnetite was generated with the formation of synthetic pyrrhotite-4C. Thus, magnetite may participate in the transformation of pyrite to pyrrhotite-4C via reactions (2)–(4). Hematite could be formed from the oxidation of pyrite by reaction (5). The possibility of reaction (6) (hematite to pyrrhotite-4C) was disapproved by a solid-phase reaction. XRD results (Figure 5) show

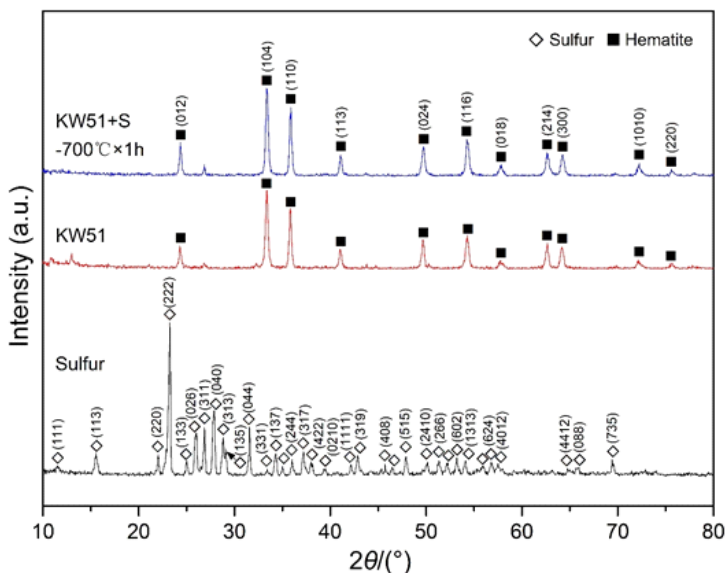
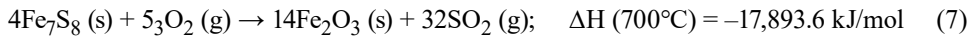


Fig. 5. XRD patterns of the raw materials and products of reaction (6)

Rys. 5. Dyfraktogramy XRD surowców i produktów reakcji (6)

that reaction (6) could not proceed at 700°C in air. By contrast, pyrrhotite-4C was completely transformed into hematite at a heating period longer than 3 h (Figure 3) according to the following reaction:



Thus, the pyrite specimen (KW30) transformed into pyrrhotite-4C at 700°C in air along two pathways: (1) pyrite → pyrrhotite-4C by reactions (1) or (2); (2) pyrite → magnetite → pyrrhotite-4C by the reactions (2) to (4). Reaction (2) is crucial to pathway (2) but not to pathway (1). Therefore, pathway (1) is more favorable than path (2). Hematite cannot be transformed into pyrrhotite-4C by reaction (6).

Conclusions

Synthetic pyrrhotite, which is formed by heating pyrite at 700°C in air, exhibits a distinct paragenetic association of minerals compared with that of natural pyrrhotite. Natural pyrrhotite and magnetite coexist in the natural pyrrhotite sample, while synthetic pyrrhotite has the paragenetic association with hematite and a small amount of pyrite and magnetite. Both are monoclinic pyrrhotite-4C (Fe_7S_8) and have minimal differences in terms of lattice parameters. Synthetic pyrrhotite-4C forms stably by heating pyrite for 0.5–2 h. Its relative content was the highest by heating for 1 h. However, pyrrhotite-4C would transform into hematite completely when heated for longer than 3 h, as was the case for pyrite and magnetite. In air, synthetic pyrrhotite-4C is formed mainly via two pathways: (1) pyrite → pyrrhotite-4C and (2) pyrite → magnetite → pyrrhotite-4C. Pathway (1) is more favorable than path (2). This transformation cannot be achieved by the reaction between hematite and sulfur.

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MINERALOGY CHARACTERISTICS, STABILITY CONDITIONS, AND FORMATION PATHWAYS OF SYNTHETIC PYRRHOTITE FORMED BY HEATING PYRITE AT 700°C**Keywords**

synthetic pyrrhotite, pyrrhotite, pyrite, hematite

Abstract

Pyrite is a sulfide mineral and is widely distributed in nature. Pyrite may transform into pyrrhotite when heated at high temperatures. In order to support processing engineering techniques and industrial applications of pyrite and pyrrhotite, it is necessary to investigate synthetic pyrrhotite, which is formed by heating pyrite in air, based on existing research. In this work, the mineralogical characteristics and stability conditions of synthetic pyrrhotite formed by heating pyrite at elevated temperatures were studied. The possible formation pathway was verified using a solid-phase reaction. X-ray-diffraction results revealed that synthetic pyrrhotite differs from natural pyrrhotite in the paragenetic association of minerals. Natural pyrrhotite and magnetite coexist in the natural pyrrhotite sample. Synthetic pyrrhotite formed by heating pyrite at 700°C for 1 h has the paragenetic association with hematite and a small amount of pyrite and magnetite. All pyrrhotite samples were monoclinic pyrrhotite-4C (Fe_7S_8) and exhibit minimal differences in terms of lattice parameters. Synthetic pyrrhotite-4C was stable under 0.5–2 h of heating at 700°C in air. It had the highest relative content by heating for 1 h. It was eventually transformed into hematite with heating periods exceeding 3 h, as was the case for pyrite and magnetite. In air, synthetic pyrrhotite-4C is mainly formed via two pathways: (1) pyrite \rightarrow pyrrhotite-4C and (2) pyrite \rightarrow magnetite \rightarrow pyrrhotite-4C. Pathway (1) is more favorable than pathway (2). This transformation cannot be achieved by the reaction between hematite and sulfur.

CHARAKTERYSTYKA MINERALOGICZNA, WARUNKI STABILNOŚCI I ŚCIEŻKI POWSTAWANIA SYNTETYCZNEGO PIROTynu UTWORZONEGO PRZEZ OGRZEWANIE PIRYTU W TEMPERATURZE 700°C**Słowa kluczowe**

pirotyn syntetyczny, pirotyn, piryty, hematyt

Streszczenie

Piryty jest minerałem siarczkowym szeroko rozpowszechnionym w przyrodzie. Piryty może przekształcić się w pirotyn podczas ogrzewania w wysokich temperaturach. W celu wsparcia technik inżynierii mineralnej i przemysłowego zastosowania piryty i pirotynu, konieczne jest zbadanie syntetycznego pirotynu w oparciu o istniejące badania, który powstaje w wyniku ogrzewania piryty w powietrzu. W pracy zbadano właściwości mineralogiczne i warunki trwałości syntetycznego pirotynu powstałego w wyniku ogrzewania piryty w podwyższonej temperaturze. Możliwą ścieżkę powstawania zweryfikowano za pomocą reakcji w fazie stałej. Wyniki dyfrakcji rentgenowskiej ujawniły,

że syntetyczny pirotyt różni się od naturalnego pirotytu w paragenetycznych asocjacjach minerałów. Naturalny pirotyt i magnetyt współlistnieją w próbce naturalnego pirotytu. Syntetyczny pirotyt powstały w wyniku ogrzewania piryty w temperaturze 700°C przez 1 godz. wykazuje asocjację paragenetyczną z hematytom oraz niewielką ilością piryty i magnetytu. Wszystkie próbki pirotytu były jednoskośnym pirotytem-4C (Fe_7S_8) i wykazują minimalne różnice pod względem parametrów sieci. Syntetyczny pirotyt-4C był stabilny w czasie 0,5–2 godzin ogrzewania w powietrzu w temperaturze 700°C. Najwyższą względną zawartość miał po ogrzewaniu przez 1 godzinę. Ostatecznie został przekształcony w hematyt z okresami ogrzewania przekraczającymi 3 godziny, podobnie jak w przypadku piryty i magnetytu. W powietrzu syntetyczny pirotyt-4C powstaje głównie dwoma metodami: (1) piryt \rightarrow pirotyt-4C i (2) piryt \rightarrow magnetyt \rightarrow pirotyt-4C. Ścieżka (1) jest korzystniejsza niż ścieżka (2). Tej przemiany nie można osiągnąć w reakcji hematytu z siarką.