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Metallurgy

Pyrolysis of copper telluride in a water vapour atmosphere

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ABSTRACT

This paper presents the results of exploratory studies on the feasibility of extracting tellurium from synthetic copper telluride and industrial tellurium–containing middlings using a vacuum-thermal method conducted in a water vapour atmosphere. It was determined that the thermal behavior of synthetic copper telluride follows an oxidation mechanism involving oxygen in a dry environment. The phase transformations occurring in the tellurium–containing industrial middlings are also comparable to those observed during oxidative-distillation roasting and vacuum-thermal processing in an inert atmosphere. The achieved degrees of extraction of copper telluride and tellurium-containing industrial middlings at a temperature of 1100 °C and a pressure of 1.3-2 kPa were 57.83 % and 94.89 %, respectively. The obtained residues are represented by copper oxide phases. At the same time, tellurium evaporates from the material and deposits on the walls of the condenser in the cold part of the reactor at temperatures below 400 °C. According to X-ray phase analysis, the condensate is represented by tellurium in the form of oxide.

Keywords: Telllurium, copper, vacuum, water vapour, evaporation

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Introduction

Tellurium is a promising material for industries such as power engineering, electrical engineering, medicine, glass production, and rubber manufacturing. Due to its low content in ores, tellurium is mainly extracted as a by-product [[1], [2], [3]]. Among the various tellurium-containing middlings (sludges, sublimates, dusts, slags, etc.), the key material is electrolytic copper sludges [[4], [5], [6]]. Due to the diverse phase composition of these sludges, traditional processing methods are complemented by new research aimed at finding more efficient technologies [[7], [8], [9], [10], [11], [12], [13]].

Industrial copper telluride obtained by the traditional method of precipitating tellurium from solution onto copper is characterized by a mixture of phases, including both non-stoichiometric ($Cu_{2-x}Te$) and stoichiometric (Cu_2Te) compositions [[14], [15], [16], [17]], as well as a small amount of impurities.

The efficiency of traditional technology to process industrial copper telluride (oxidative-alkaline leaching) is about 80 % according to [16]. Therefore, developing a more effective method is an important direction. Studies are known that focus on improving the traditional method [[16], [18], [19]], as well as developing alternative pyrometallurgical approaches.

Pyrometallurgical methods have long been uncommon in both industrial practice and scientific research. This is since the decomposition of Cu_2Te is possible only at temperatures above 2704 °C [[20], [21]]. A significant reduction in temperature can be achieved by conducting the process under low pressure.

However, copper telluride has a low dissociation pressure: at 1780 °C, it is 0.7 kPa. Therefore, direct tellurium extraction via Cu_2Te decomposition is theoretically impossible.

Consequently, existing research on pyrometallurgical methods focuses on finding optimal conditions for tellurium extraction using reagents that lower the decomposition temperature of copper telluride. For example, paper [22] proposed using elemental sulfur, which replaces tellurium in copper telluride.

The technology is two-stage and involves pre-sulfidation of the tellurium–containing middlings followed by vacuum-thermal sublimation of elemental tellurium. The efficiency of tellurium extraction is 97 %. In paper [23], air oxygen was proposed as an oxidizer. This technology is also two-stage. Tellurium is extracted from the tellurium–containing middlings in oxide form, which is then subjected to thermal reduction. The efficiency of tellurium extraction is 98 %. Paper [24] considered the possibility of extracting tellurium in an inert atmosphere using the oxygen contained in the material itself. Tellurium was also extracted in oxide form, with an extraction efficiency of about 99 %.

These papers [[23], [24]] were based on the more favorable vapor pressure of tellurium oxide (TeO_2), which at 733 °C is about 0.02 kPa [[25], [26]]. However, it was found that during both oxidative distillation roasting and vacuum thermal processing, up to the evaporation temperature, tellurium oxide interacts with copper oxide. As a result, copper orthotellurate (Cu_3TeO_6) is formed, which is stable up to 880 °C [27].

It is known that in a water vapour atmosphere, the volatility of tellurium oxide increases due to the formation of the more volatile $TeO(OH)_2$ [[28], [29]], which upon condensation converts to tellurium dioxide [30].

In this regard, we conducted a study aimed at evaluating the effect of water vapour on tellurium extraction from tellurium–containing middlings. The results of the work are presented in the current article.

Experimental part. Materials

Synthetic Copper Telluride.

The study used synthetic copper telluride obtained by direct melting of the starting components in an evacuated quartz ampoule. For the synthesis, electrolytic copper chips (99.99 %) and elemental tellurium powder (99.98 %) were taken in stoichiometric amounts (49.92 wt.% % Cu and 50.08 wt.% % Te). The synthesis temperature was 1200 °C, with a heating rate of 2 °C/min up to the target temperature. The synthesis duration was 6 hours. The obtained alloys were slowly cooled in the furnace after the set holding time.

Phase analysis of the material was conducted using the ICDD PDF-2 database (relies 2023) and corroborated with literature data [[31], [32]]. Semi-quantitative phase analysis (Figure 1) showed that the main portion of the alloy consisted of the Cu_7Te_4 ($Cu_{1.75}Te$) phase at 85.1 %, while the $Cu_{0.664}Te_{0.336}$ ($Cu_{1.91}Te$) phase was present at 14.9 %. According to the literature, the primary phase corresponds to Cu_2Te.

Tellurium-Containing Middling.

Industrial copper telluride obtained at Kazakhmys Smelting LLP (Republic of Kazakhstan) was used as a tellurium-containing middling.

Copper telluride is an agglomerated material of malachite color, odorless, with a moisture content of 3%. The material composition is presented in Table 1. X-ray phase analysis (Figure 2) revealed a significant fraction of amorphous halo due to scattering from disordered phases. The crystalline part is represented by phases of non-stoichiometric copper telluride (Cu_7Te_4 – PDF 00-057-0196, $Cu_{1.79}Te$ – PDF 01-082-9896) and copper hydroxysulfates ($Cu_5(SO_3)_2(OH)_6 \cdot 5H_2O$ – PDF 00-041-0007, $Cu_3(SO_4)(OH)_4$ – PDF 00-007-0407, $Cu_4(SO_4)(OH)_6 \cdot H_2O$ – PDF 01-083-1410, $Cu_6SO_4(OH)_6$ – PDF 00-043-1458). The presence of the latter can be explained by insufficient washing of copper telluride from the $CuSO_4$ solution after the operation of tellurium cementation on copper from tellurous acid.

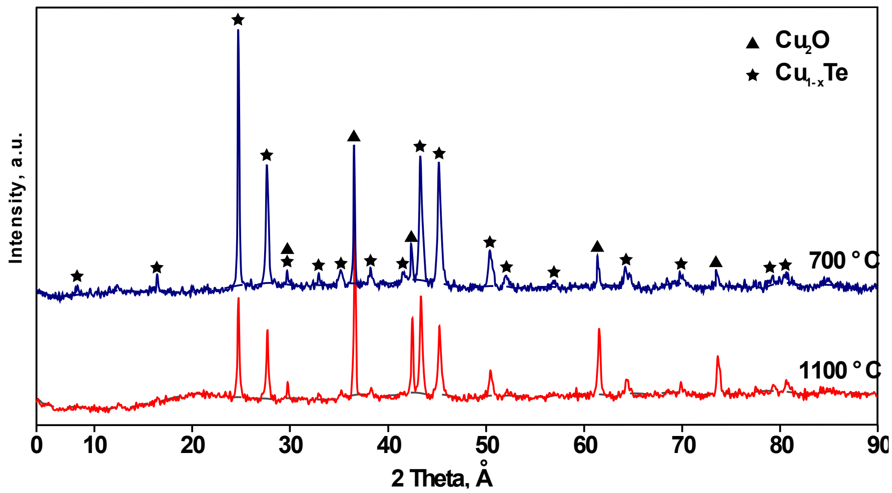


Figure 1 - Diffractogram of synthetic copper telluride

Table 1. Composition of the tellurium-containing middling

Elemental composition, wt. %									
O	Al	Si	S	Cl	Cu	As	Te	Se	Pb
31.38	0.02	0.05	2.31	0.20	42.45	0.12	23.42	0.03	0.02

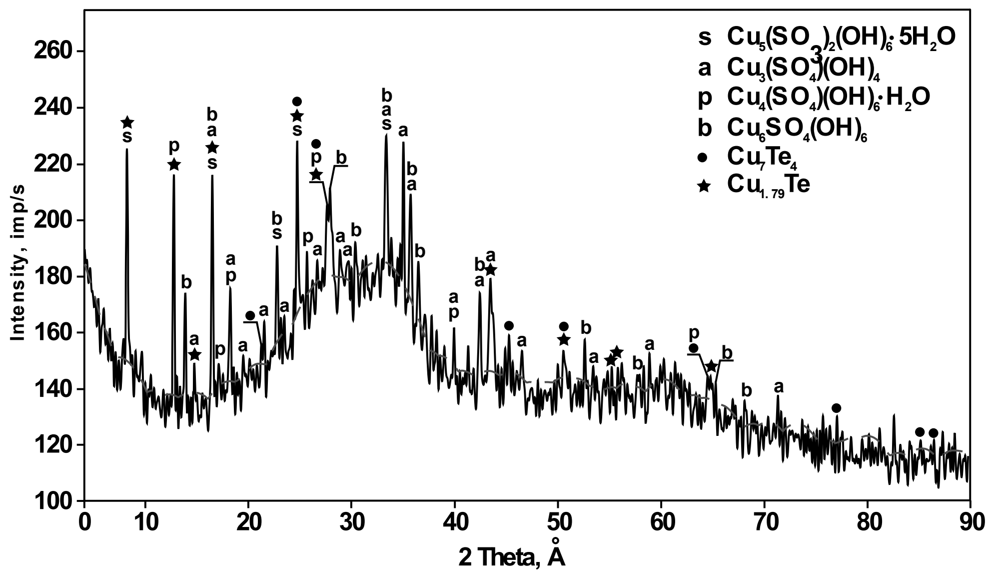


Figure 2 – X-ray diffraction pattern of the tellurium–containing middling from Kazakhmys Corporation L

Methodology

A laboratory setup with a horizontally arranged reactor was used for the study (Figure 3).

The setup consists of a Nabertherm electric furnace with a B–180 controller, a 2HB3-5DM UHL4 vacuum pump, and a quartz reaction vessel, in which a boat containing the sample of predetermined mass was placed. Additionally, a detachable porcelain condenser was mounted over the boat to collect the condensed material. A chromel–alumel thermocouple (thermoelectric transducer DTPK021-1.2/0.7) with a single-channel microprocessor-based measuring controller TRM1 was used to control the temperature in the reaction zone. The pressure was measured with a DCP 3000 vacuum gauge (Vacuubrand, Germany) with a VSP 3000 sensor (accuracy ±10 Pa). The sample was weighed before and after the experiment with a PA214C analytical balance (Ohaus-Pioneer) with an accuracy of ±0.1 mg.

Argon was used as the carrier gas saturated with water vapor in a flask filled with distilled water. The flask was placed in a thermostat. Argon flow was controlled using an RS-3A rotameter. Two desiccators were installed, represented by Tishchenko bottles filled with alumogel, to dry the exhaust gases.

The experimental procedure was as follows. The copper telluride sample was loaded into an alumina boat. The boat was then placed into the detachable (longitudinal) alumina condenser. The condenser was in turn placed into the quartz reactor. The prepared reactor was placed in the preheated furnace so that the sample was located in the isothermal zone. The reactor was connected to the vacuum system and the water vapor supply system. The vacuum pump was turned on, and the argon flow was set. The water vapor flow into the reaction chamber was regulated by adjusting the thermostat temperature. The water flow rate was determined by weighing the flask before and after the experiment. The start of the experiment was defined as the moment when the target pressure and temperature values, recorded by the monitoring thermocouple, were reached. The gas supply and evacuation systems were turned off at the end of the experiment. The reactor was removed from the furnace and allowed to cool in air. The obtained calcination products were weighed and analyzed.

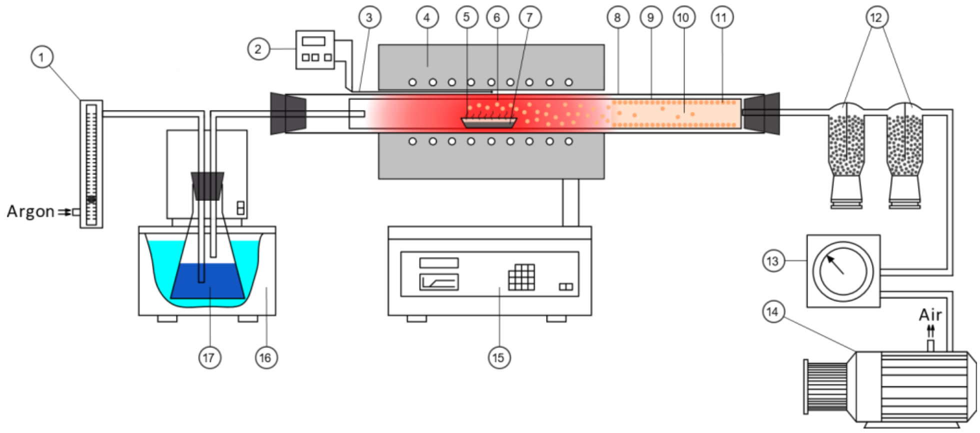
Characterization. The study of the material composition was performed by X–ray fluorescence analysis using a wavelength-dispersive spectrometer Axios 1kW (PANalytical, Netherlands) with an accuracy of ±5 %.

For phase composition identification, X–ray diffraction analysis was performed using a D8 Advance diffractometer (Bruker, Germany), Cu-Kα radiation. The phase composition was determined using the ASTM database (reference database of diffraction data PDF-2 rel. 2023 of the International Centre for Diffraction Data (ICDD, USA)).

Results and discussion

Experiments with the synthetic material were performed under the following conditions: temperature 700–1100 °C, pressure 1.3 kPa, duration 10–60 min, humidity 0.5–1 %. The degree and rate of oxidation were determined based on CuO, where O = 20.2 %. The results are presented in Table 2.

As can be seen, exposure of synthetic copper telluride to water vapor significantly increases the degree of tellurium evaporation (up to 58%) compared to the process conducted in an inert atmosphere (0.5–2 %) [33]. A positive effect on tellurium extraction is observed with increasing duration and temperature of the process, while increasing the argon flow rate from 5.21 to 32.7 L/h has little effect on the degree of tellurium evaporation from synthetic copper telluride. At the same time, the degree of oxidation of copper telluride itself shows an almost linear dependence on the duration and temperature of the process. It should be noted that decreasing the water vapor flow rate promotes longer interaction between copper telluride and oxygen.



1 – rotameter; 2 – temperature controller in the reaction zone; 3 – control thermocouple;
4 – electric furnace; 5 – boat; 6 – isothermal zone; 7 – sample; 8 – reactor; 9 – detachable condenser;
10 – condensation zone; 11 – condensate; 12 – desiccators; 13 – aneroid barometer; 14 – vacuum pump;
15 – furnace controller; 16 – thermostat; 17 – flask with distilled water

Figure 3 - Installation diagram of the pyrolysis setup in a water vapor atmosphere

Table 2 – Dependence of tellurium extraction from synthetic copper telluride and the degree of copper telluride oxidation on key process parameters

Experiment No.	Temperature, °C	Ar Flow Rate, L/h	Time, min.	Te Content in Residue		Te Evaporation Degree, %	Cu_2Te Oxidation Degree, %
				%	g		
1	700	13.0	60	40.76	1.2746	11.4857	32.8218
2	700	13.0	30	41.26	1.3071	9.2280	26.6337
3	700	13.0	10	43.99	1.3600	5.5529	20.1485
4	900	13.0	60	40.60	1.1798	18.0669	35.0495
5	900	13.0	30	41.29	1.2434	13.6552	28.1040
6	900	13.0	10	43.23	1.3103	9.0069	22.6436
7	1100	13.0	60	28.34	0.7004	51.3615	38.7178
8	1100	13.0	45	30.42	0.8223	42.8991	34.5050
9	1100	13.0	30	34.58	0.9548	33.6977	31.6337
10	1100	13.0	10	41.63	1.2260	14.8638	24.2475
11	1100	32.7	60	30.93	0.8077	43.9115	35.00
12	1100	5.21	60	24.57	0.6073	57.8266	56.58

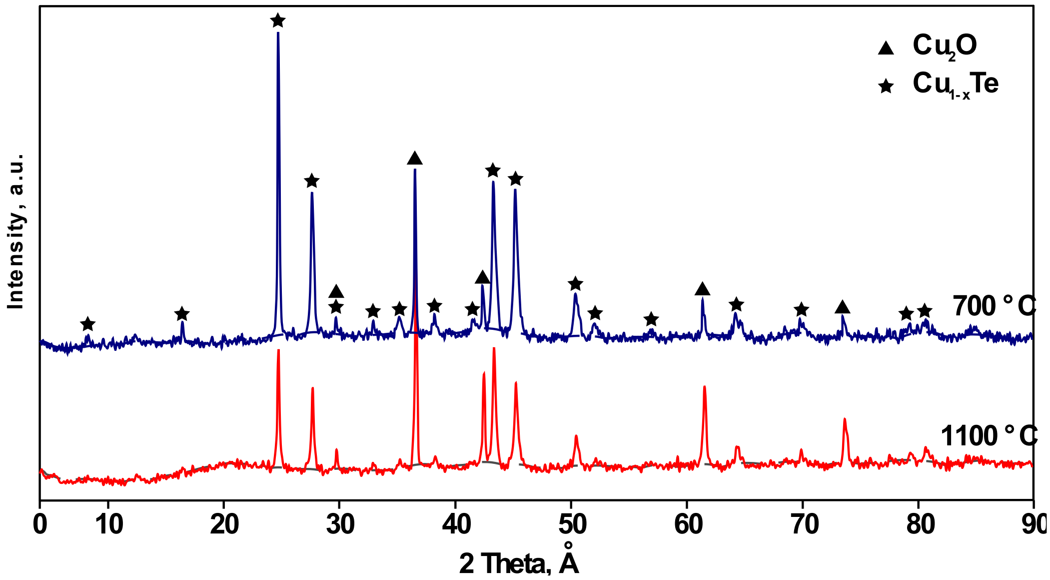


Figure 4 – Diffractograms of residues obtained at 700 °C and 1100 °C

Based on the experimental data, the dependence of the average rate of tellurium evaporation and copper telluride oxidation on temperature is described by equations (1) and (2), respectively:

$$\lg V = -2{,}103.3/T - 2.0838, \quad \text{where } E_{app.\ act.} = 40.27 \text{ kJ/mol} \qquad (1)$$

$$\lg V = -234.56/T - 3.5017, \quad \text{where } E_{app.\ act.} = 4.49 \text{ kJ/mol} \qquad (2)$$

Analysis of the phase composition of the obtained residues revealed, among the crystalline phases, the presence of copper oxide and unreacted copper telluride in varying ratios (from 18.9 to 58.0 wt. % Cu_2O). A typical X-ray diffraction pattern is shown in Figure 4. As seen in the figure, the diffractogram contains an amorphous halo, indicating the presence of compounds with a disordered structure in the residues. Based on literature data [[24], [34], [35]], it can be assumed that the amorphous phase consists of copper tellurates and/or tellurites formed during the interaction of oxidized copper and tellurium, as well as tellurium oxide. The phase composition of the residues is presented in Table 3.

Table 3 – Phase composition of residues

Experiment No.	Content of Crystalline Phases, wt. %		Degree of Amorphousness, %
	Cu_2O	$Cu_{2-x}Te$	
1	19.8	80.2	34.4
2	42.7	57.3	39.4
3	56.4	43.6	40.6
4	32.0	68.0	49.3
5	18.9	81.1	30.6
6	22.4	77.6	34.8
7	58.0	42.0	39.4
8	38.5	61.5	37.5
9	42.2	57.8	36.6
10	25.9	74.1	35.7
11	44.7	55.3	31.5
12	46.6	53.4	37.4

Table 4 – Dependence of tellurium extraction from industrial copper telluride on main process parameters

Temperature, °C	Ar Flow Rate, L/h	Time, min.	Te Content in Residue		Te Evaporation Degree, %
			%	g	
700	13.0	60	25.075	0.52	0
700	13.0	30	24.879	0.52	0
700	13.0	10	23.71	0.52	0
900	13.0	60	7.201	0.14	71.87
900	13.0	30	15.13	0.31	39.49
900	13.0	10	21.896	0.47	8.74
1100	13.0	60	6.228	0.09	83.21
1100	13.0	30	10.979	0.16	67.91
1100	13.0	10	22.031	0.39	22.65
1100	32.7	60	6.552	0.09	81.67
1100	5.21	60	2.211	0.03	94.89

Experiments with the tellurium–containing middling were performed under the following conditions: temperature 700–1100 °C, pressure 2 kPa, duration 10–60 min, humidity 0.5–1 %. The results are presented in Table 4 and Figure 5.

As follows from the obtained data, at 700 °C no tellurium evaporation from the industrial telluride occurs, which is associated with the formation of the most stable copper orthotellurate Cu_3TeO_6, stable up to 900 °C. Cu_3TeO_6 is the decomposition product of phase X, the identification of which is complicated due to the absence of the necessary compound card in the PDF-2 database (2023 edition). Considering the probable composition of phase X – $Cu_{0.37}Te_{0.26}O_{0.76}S_{0.084}$ – and the absence of formation of the aforementioned phases during oxidation of the synthetic telluride, the following can be assumed. Phase X is formed during the oxidation of the industrial telluride, due to the interaction of decomposition products of copper hydroxysulfates with oxidized copper and tellurium. This phase subsequently decomposes to Cu_3TeO_6. In contrast, during the oxidation of the synthetic material, the tellurium extraction process proceeds without the formation of the sulfur-containing phase X and, consequently, without formation of copper orthotellurate, following the oxidation mechanism described in [34]. At 900 °C, the formation of orthotellurate is observed within the first 10 min and is completely decomposed over the next 20 min of the experiment. At 1100 °C, only crystalline copper oxides are identified in the obtained residues. The maximum tellurium extraction reaches 94.89 % under the following conditions: temperature – 1100 °C, pressure – 15 mm Hg, process duration – 60 min, carrier gas flow rate – 5.21 L/h, humidity – 0.7 % (with evaporated water temperature 27 °C).

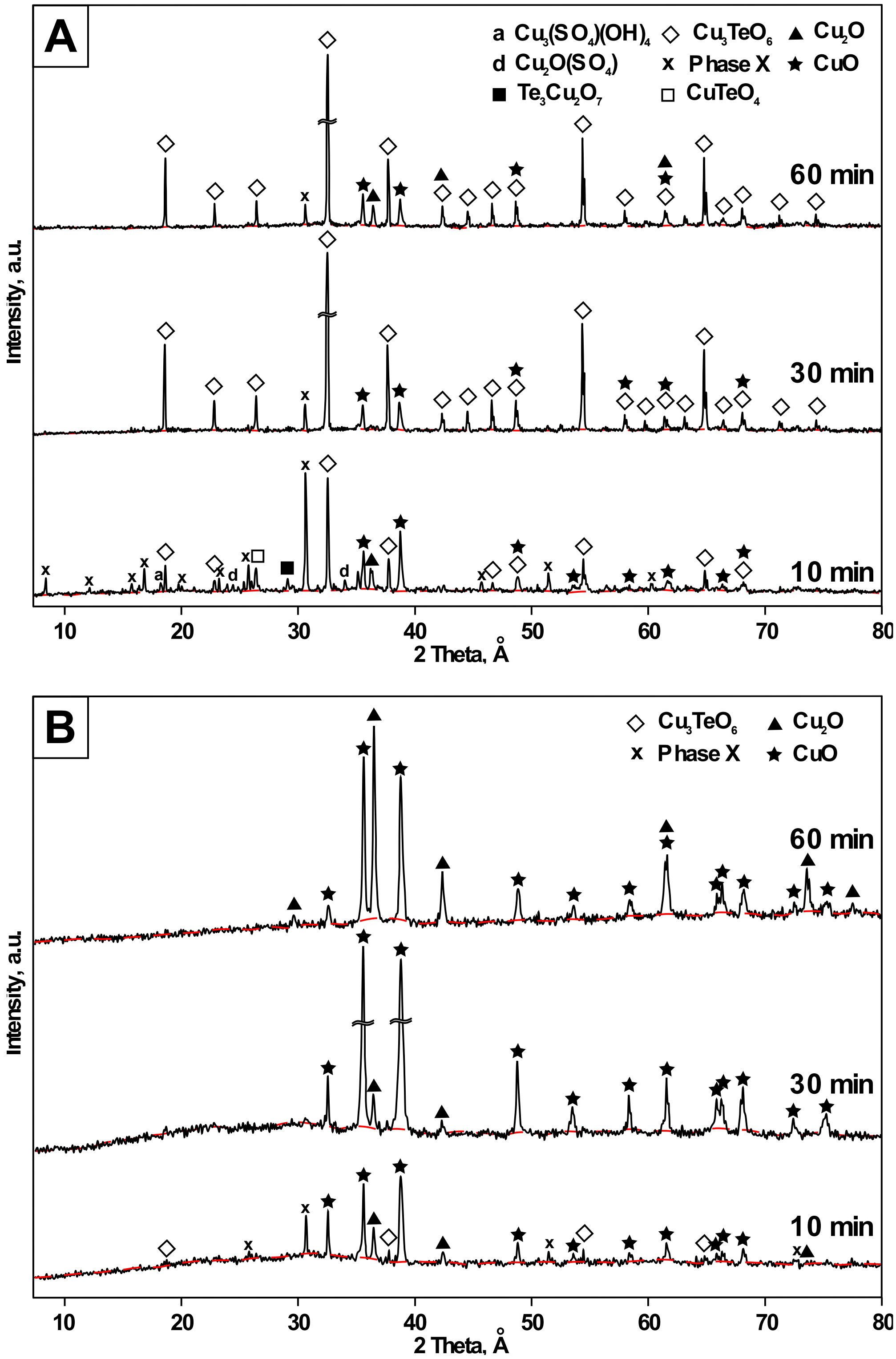


Figure 5 – Phase composition of residues obtained at 700 (a) and 900 (b) °C

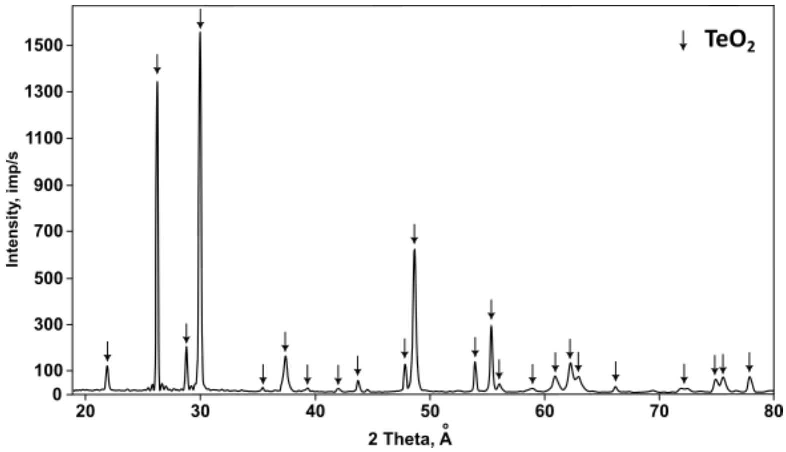


Figure 7 – XRD of the tellurium-containing condensate

Based on the experimental data, the dependence of the average tellurium evaporation rate on temperature is described by equation (3):

$$lgV = -10{,}184/T + 4.5259,$$
$$\text{where } E_{app.\ act.} = 194.96 \text{ kJ/mol} \qquad (3)$$

During the roasting process, tellurium evaporated from the tellurium-containing by-product and deposited on the walls of the corundum condenser. The obtained condensate is a dense fine-crystalline white powder (Figure 6). Determination of the deposition conditions established that the vapor–gas phase condensed in the "cold" part of the reactor at temperatures below 600 °C. X-ray diffraction analysis (Figure 7) showed that the deposited condensate is represented by a monophasic tellurium oxide (TeO_2). Further processing of such a condensate to obtain elemental tellurium does not present technological difficulties. It can be carried out by carbothermal reduction in vacuum at moderate temperatures.

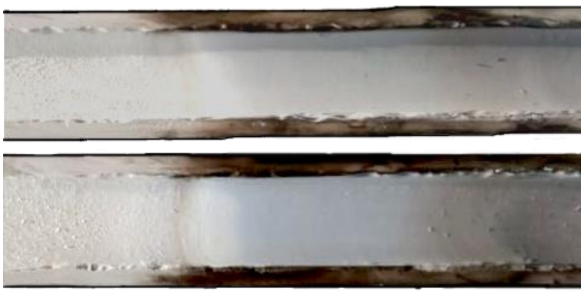


Figure 6 – Photo of the deposited condensate

Conclusion

Thus, the conducted set of studies demonstrated the feasibility of tellurium extraction from synthetic copper telluride and industrial tellurium-containing middling using a vacuum-thermal method conducted in a water vapour atmosphere. The thermal behavior of synthetic copper telluride follows the oxidation mechanism known from literature data. The behavior of the tellurium-containing middling is similar to that previously described for oxidative-distillation roasting and vacuum-thermal processing in an inert atmosphere. The achieved tellurium extraction values for synthetic copper telluride and tellurium-containing middlings were 57.83 % and 94.89 %, respectively, at a temperature of 1100 °C and pressure of 1.3–2 kPa. Incomplete oxidation of the synthetic material is apparently due to the insufficient amount of oxygen supplied by the water vapor flow. The obtained condensate is represented by the TeO_2 phase. The tellurium-containing condensate serves as a raw material for the production of elemental tellurium using well-known technologies, such as carbothermal reduction.

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Conflict of interest. The corresponding author declare that there is no conflict of interest.

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Мыс теллуридінің сулы бу атмосферасындағы пиролизі

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Қабылданды: *3 қазан 2025*

ТҮЙІНДЕМЕ
Жұмыста су буының атмосферасында жүргізілетін вакуумды-термиялық әдісті қолдана отырып, синтетикалық мыс теллуридінен және құрамында теллур бар өнеркәсіптік ортадан теллур алу мүмкіндігін анықтау бойынша барлау зерттеулерінің нәтижелері берілген. Синтетикалық мыс теллуридінің жылулық әрекеті құрғақ ортада оттегінің қатысуымен тотығу механизміне сәйкес келетіні анықталды. Құрамында теллур бар аралық өнімде орын алатын фазалық өзгерістер де тотығу-дистилляциялық күйдіру және инертті атмосферадағы вакуум-термиялық үрдіс кезінде сипатталғандармен салыстыруға болады. 1100 °C температурада және 1,3-2 кПа қысымда мыс теллуридті және құрамында теллур бар өнеркәсіп өнімдерін алу дәрежесінің қол жеткізілген мәндері сәйкесінше 57,83 және 94,89 %-ды құрады. Алынған қалдықтар мыс оксидтерінің фазаларымен ұсынылған. Бұл жағдайда теллур материалдан буланып, конденсатордың қабырғаларында реактордың суық бөлігінде 400 °C-тан төмен температурада тұндырылады. Рентгендік фазалық талдауға сәйкес конденсат теллурмен оксид түрінде ұсынылған.

Түйін сөздер: теллур, мыс, вакуум, су буы, булану.

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Пиролиз теллурида меди в атмосфере водяного пара

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АННОТАЦИЯ
В работе приведены результаты поисковых исследований по определению возможности извлечения теллура из синтетического теллурида меди и промышленного теллурсодержащего промпродукта вакуум-термическим способом, проводимым в атмосфере водяного пара. Определено, что термическое поведение синтетического теллурида меди подчиняется механизму окисления с участием кислорода в сухой среде. Фазовые преобразования, происходящие в теллурсодержащем промпродукте также сопоставимы с описанными при окислительно-дистилляционном обжиге и вакуум-термическом процессе в инертной атмосфере. Достигнутые значения степени извлечения теллурида меди и теллурсодержащего промпродуктов при температуре 1100 °C и давлении 1,3-2 кПа составляли 57,83 и 94,89 %, соответственно. Полученные остатки представлены фазами оксидов меди. При этом теллур испаряется из материала и осаждается на стенках конденсатора в холодной части реактора при температурах ниже 400 °C. По данным рентгенофазового анализа конденсат представлен теллуром в виде оксида.

Ключевые слова: теллур, медь, вакуум, водяной пар, испарение.

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