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Behavior of Technical Copper Telluride During Vacuum Thermal Treatment

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ABSTRACT

This study investigates phase transformations in copper-tellurium-containing middlings during vacuum-thermal processing at pressures of 0.066 and 13.3 kPa. It was found that at 0.066 kPa, industrial copper telluride undergoes oxidation through intermediate compounds to Cu_3TeO_6 , which decomposes at 900°C via CuTeO_3 into CuO and TeO_2 . At 13.3 kPa, the formation of CuTeO_3 through the Cu_3TeO_6 was not observed. Technological tests showed the feasibility of tellurium recovery by the vacuum-thermal method in an inert atmosphere without additional reagents. It was found that reduced pressure and elevated temperature positively influenced the degree of tellurium transfer into the condensate. The highest recovery rates were achieved at 1100°C: 98.70% at 0.066 kPa and 96.79% at 0.133 kPa. The residual Te content in residues amounted to 0.72% and 1.62%, respectively. The findings expand scientific understanding of the thermal behavior of industrial copper telluride under vacuum conditions and provide a foundation for developing a new environmentally friendly method of processing tellurium-containing middlings. The creation of new technologies enhances the extraction efficiency of tellurium while reducing ecological risks.

KEYWORDS

Copper telluride; vacuum treatment; decomposition mechanism; tellurium; recovery

Introduction

Tellurium is a chalcogen and a rare element used as a promising material for diverse industries, including solar energy, electrical engineering, electric power, medicine, glass, rubber, and metallurgy. In nature, tellurium is found both in its native state and as part of various telluride minerals (Chizhikov and Shchastlivyi 1970; Paterson 1990; Sahu et al. 2020). However, the independent production of tellurium from tellurium-containing minerals is complicated by their low content and complex mineralogical association with non-ferrous and noble metals (Corchado-Albelo et al. 2024). Tellurium is mainly extracted as a by-product, since during technological processes it is concentrated in sludge, dust, sublimation, skimmings, drains, and slag (Chibesa, Nakhaei, and Alagha 2025; Haque 1987; Jha 1987; Wang and Forssberg 1990). Therefore, the issue of processing tellurides is an important direction in modern research (Rivera Li Kao and Garbers-Craig 2024; Rodríguez-Chávez, Fuentes-Aceituno, and Nava-Alonso 2022; Tian and Liu 2022; Villa Gomez et al. 2024; Xu et al. 2018).

The primary raw material source of tellurium is copper electrolysis sludge, which serves as a collector of precious metals and chalcogens (Chen et al. 2015; Koizhanova et al. 2024; Liu et al. 2020; Mahmoudi et al. 2020). Chalcogens, such as selenium and tellurium, are present as compounds with copper, silver, and gold. Owing to the complexity and diversity of the chemical and phase composition of sludge, a wide range of processing technologies, including new methods, have been developed (Ding et al. 2017; Kenzhaliyev et al. 2018, 2019; Liu et al. 2020; Mahmoudi et al. 2020; Mastuyugin and Naboichenko 2012; Xiao et al. 2018; Xing and Lee 2017).

Tellurium can be obtained from copper-electrolyte sludge by combining hydrometallurgical, pyrometallurgical, electrometallurgical processes (Chizhikov and Shchastlivyi 1970; Hoffmann 1989; Mastuyugin et al. 2013). Existing processes for producing branded tellurium involve several technological and technical difficulties owing to multi-stage processes, high reagent consumption, and the formation of large volumes of heavy-metal-containing wastewater. Therefore, some facilities accumulate and/or sell copper telluride, which is a tellurium precipitation product from copper-electrolyte sludge dehydration solutions. Industrial copper telluride is a mixture of phases with both stoichiometric (Cu_2Te) and non-stoichiometric (Cu_{2-x}Te) compositions (Nitsenko et al. 2020, 2021; Shibasaki, Abe, and Takeuchi 1992; Wang 2011; Xu et al. 2020) and contains small amounts of impurities of other elements and compounds.

Traditionally, tellurium-containing middling products undergo redox-alkaline leaching with NaOH addition in the first stage (Hoffmann 1989; Kenzhaliyev et al. 2019; Mastuyugin et al. 2013). This process concentrates tellurium in solution as sodium tellurite (Na_2TeO_3), while copper remains in the residue. The solution is then electrolyzed, and copper is extracted from the residue. According to Xu et al. (2020), the classical method achieves a tellurium-leaching efficiency of approximately 80%, which is attributed to the formation of insoluble CuTeO_3 . Tellurium is a significant element in high-tech industries and advanced technologies; therefore, its efficient and environmentally safe extraction from copper production middlings is especially relevant.

Zhang et al. (2025) provided an overview of methods developed for processing industrial tellurides. However, as

the authors noted, these methods are mainly at the laboratory-research stage and have not been applied in industrial practice.

In addition to those cited by Zhang et al. (2025), it is worth noting work aimed at improving the traditional hydrometallurgical method to increase tellurium extraction from the middling product (Xu et al. 2020, 2020).

Xu et al. (2020) compared a classical atmospheric leaching method with a proposed autoclave-leaching method, achieving over 95% tellurium dissolution using the latter. After autoclave leaching, the precipitate contained only crystalline Cu_2O , allowing its return to copper production. The tellurium-containing solution is neutralized with sulfuric acid to precipitate tellurium as TeO_2 , yielding a residue with 95% TeO_2 . Using H_2O_2 in a two-stage redox-alkaline leaching process (Xu et al. 2020) achieved a 93% tellurium recovery rate. Overall, the end-to-end recovery of tellurium reached nearly 90%, with the residue containing 96% TeO_2 . The authors also proposed transferring the tellurium-containing residue for the electrolytic production of elemental tellurium.

When studying the described methods in more detail, it should be noted that improving the classical method by using an autoclave or adding H_2O_2 increases the leaching efficiency but does not address several traditional shortcomings of hydrometallurgical methods, including the use of water, multi-stage processes, reagent consumption, need for solution neutralization and disposal, and lengthy processing times.

The pyrometallurgical method is an alternative to hydrometallurgical methods. However, according to the temperature dependences of the vapor pressure of tellurium over solid Cu_2Te (Dutchak, Korenchuk, and Korenchuk 1975; Weissburd 1996), the decomposition of the compound into copper and tellurium is possible only above 2704°C (Nitsenko et al. 2023). Owing to this drawback, pyrometallurgical methods are not widely used in research or industrial practice.

An effective way to reduce the process temperature is to conduct it at low pressure, which improves the energy efficiency. In addition, vacuum processing enhances workplace safety and reduces the environmental impact. This is particularly important when handling raw materials containing toxic elements such as tellurium. Tellurium compounds (hydrogen telluride, telluric acid, its salts, sodium tellurite, and tellurium oxide) exhibit pronounced toxicity, including irritating respiratory tract irritation and the potential to cause chronic poisoning. Recent studies (Chizhikov and Shchastlivyi 1970; Kolesnikov et al. 2024; Nassar et al. 2022; Nuss 2019; Presentato et al. 2019; Qin et al. 2017) have shown that the increase in tellurium content in the environment is anthropogenic and linked to industrial sources, including metallurgical operations. Stricter environmental protection requirements have stimulated the development of low-pressure pyrometallurgical methods. Conducting the process in sealed equipment with the condensation-based capture of tellurium-containing compounds significantly reduces the environmental risks associated with tellurium toxicity.

Due to the low dissociation pressure of liquid telluride (0.7 kPa at 1780°C), obtaining tellurium by decomposing Cu_2Te under such conditions using a vacuum-thermal method appears unfeasible. Therefore, current research focuses on alternative methods involving oxygen or sulfur to eliminate

the thermal effect on the telluride environment and convert tellurium into its oxide or elemental form.

Li et al. (2022) proposed extracting tellurium from copper telluride using a two-stage process: preliminary sulfidation of the raw material followed by vacuum-thermal sublimation of tellurium from the resulting product. Elemental sulfur powder was used as the sulfidiser and mixed with copper telluride. The resulting charge was then briquetted. Sulfidation was performed at atmospheric pressure in an inert atmosphere. The sublimation of tellurium from the sulfide material was conducted at 10–20 Pa. This low pressure enabled the extraction of up to 97% of tellurium at 650°C with a purity of 96.37%. All copper in the vacuum residue was in sulfide form.

Previously, we tested a method of oxidative distillate roasting of copper telluride (Nitsenko et al. 2022) for the removal of tellurium dioxide from a middling product under laboratory conditions. It was shown that at a temperature of 1000°C (pressure 0.67 kPa, oxidant flow rate (air) $2.2 \times 10^{-2} \text{ m}^3/\text{m}^2 \cdot \text{s}$, roasting duration 60–90 min), the tellurium extraction rate reached 90–93%. When the temperature was increased to 1100°C (firing duration 60 min), the degree of tellurium extraction into the condensate was acceptable from a technological point of view (98%). One of the decomposition products is a copper oxide alloy, which forms the basis of the residue. The second product is the oxide form of tellurium, which evaporates and then condenses in crystalline form in the cold zone of the condenser. The main component of the condensate phase was tellurium oxide, which can be processed into elemental chalcogen via thermal reduction or electrolysis in a single operation.

In developing the oxidation – distillation method (Nitsenko et al. 2022), similar temperature-dependent mass-loss curves were observed in both oxidizing and inert atmospheres. At 1100°C, the final mass loss of industrial copper telluride was approximately 21% in both cases, with the system pressure at 92 kPa. The likely cause of the mass loss of copper telluride under an inert atmosphere, beyond dehydration, is the presence of oxidizers within the material itself.

In this regard, we studied the thermal behavior of industrial copper telluride under vacuum-thermal treatment in an inert atmosphere. The data indicates that copper telluride can be processed by extracting tellurium in its oxidized form without using additional reagents.

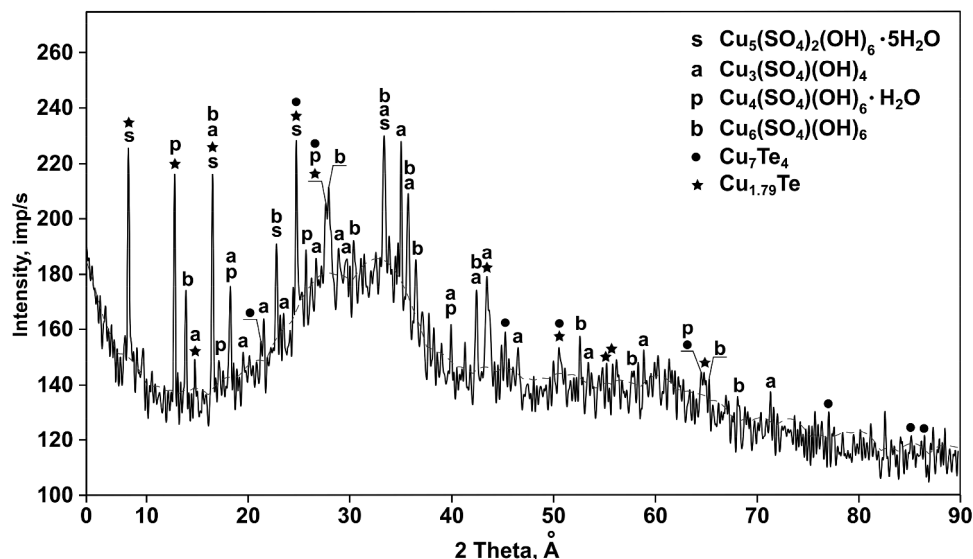
Materials and methods

Materials

The tellurium-containing middling product used in this study was obtained from copper-electrolyte sludge at the Kazakhmys Melting LLP refinery (Republic of Kazakhstan). The middling product was an odorless, pelletized malachite material with 3% humidity. The elemental composition of the copper telluride is presented in Table 1. Based on the X-ray phase analysis results (Figure 1), the presence of a large proportion of amorphous halos owing to scattering from disordered phases was established. The crystalline portion was represented by copper telluride phases of non-stoichiometric composition (Cu_7Te_4 – PDF 00–057–0196, $\text{Cu}_{1.79}\text{Te}$ – PDF 01–082–9896) and copper hydroxosulfates ($\text{Cu}_5(\text{SO}_3)_2(\text{OH})_6 \cdot 5 \text{H}_2\text{O}$ – PDF 00–041–0007,

Table 1. Composition of the tellurium-containing middling product.

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

**Figure 1.** X-ray diffraction pattern of the tellurium-containing industrial product from Kazakhmys corporation LLP.

$\text{Cu}_3(\text{SO}_4)(\text{OH})_4$ – PDF 00–007–0407, $\text{Cu}_4(\text{SO}_4)(\text{OH})_6 \cdot \text{H}_2\text{O}$ – PDF 01–083–1410, $\text{Cu}_6\text{SO}_4(\text{OH})_6$ – PDF 00–043–1458). The presence of the latter could be explained by the insufficient washing of copper telluride from the CuSO_4 solution after the cementation of tellurium on copper from telluric acid. As a result, under the influence of humidity, the presence of sulfate ions in the material, and storage conditions, processes occurred over time that contributed to the formation of hydroxosulfates and oxidation of copper telluride.

Prior to testing, the copper telluride sample was ground to a size of less than 0.315 mm. The material was not pre-dried.

Argon (99.993% purity) was used to flush the internal system of the vacuum installation and for conducting the experiments.

Methodology

Two laboratory units were used for the study: a vertical reactor (Figure 2) was used to determine the mechanism of weight loss when copper telluride was heated under non-isothermal conditions, and a horizontal reactor arrangement (Figure 3) was used to study the thermal behavior of copper telluride during pyrolysis under isothermal conditions. The units consisted of an RT 50/250/13 tube electric furnace (Nabertherm, Germany) with a V–180 controller and gas evacuation system. The furnace was then placed in the reactor. The isothermal zone of the furnace was determined by multipoint temperature measurements along the length of the chamber at 1-cm increments. A 2NVR–5DM UHL4 vacuum pump (Vacuum Mash, Russia) was used to create a vacuum in the system. A leak valve maintained constant pressure in the system near the pump. The pressure was measured with a DCP

3000 vacuum gauge (Vacuubrand, Germany) using a VSP 3000 sensor (error ± 10 Pa). During the experiments, the pressure deviation in the reactor from the specified value did not exceed 15 Pa. A filter was used to trap particles not settled in the reactor or retort outlet. A Chromel – Alamel thermocouple (DTPK021–1.2/0.7 thermoelectric transducer) with a TRM1 single-channel microprocessor meter regulator controlled the temperature of the reaction zone.

Determination of the weight loss mechanism due to heating under non-isothermal conditions

The reactor in the unit consisted of two parts: the lower part was made of alloy steel and placed in an electric furnace, and the upper part was made of heat-resistant glass. A crucible with a copper telluride hanger was installed in the hollow suspension inside a steel retort. A thermocouple junction was located inside the suspension at the level of the linkage in the crucible. The suspension rested on the scale of the weight-loss measurement system located in the upper part of the retort. Part of the retort was connected outside the high-temperature zone using a rubber seal. The lower and upper parts of the retort were separated by screens to reduce heat flow from the high-temperature zone. The upper part of the retort housed the pressure-measurement system, channels for gas evacuation and argon filling, and thermocouple end outlets. The weight-, pressure-, and temperature-measurement systems had signal outputs to a multipoint potentiometer, with measurements recorded on a diagram tape.

The alloy load was then placed in a quartz crucible for the experiments. It was mounted on a suspension with the retort disconnected from the heating zone. The lower part of the

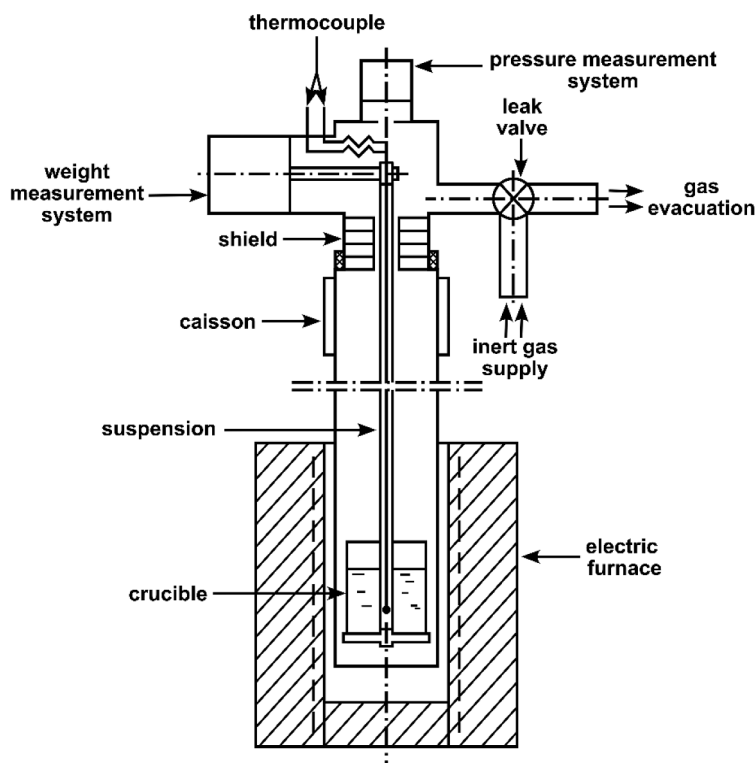


Figure 2. Schematic of the installation for continuous thermogravimetry.

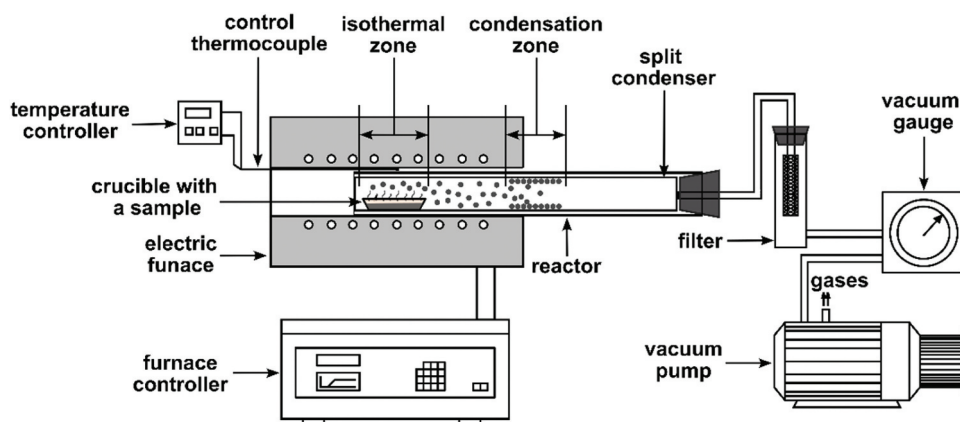


Figure 3. Horizontal vacuum unit with a split condenser.

retort was connected to the upper part. The pressure specified by the experimental conditions was established after three stages of flushing the system with argon using a vacuum pump. The lower part of the retort was then placed in the isothermal zone of the electric furnace, and furnace heating was initiated. The material weight loss, temperature change, and pressure change in the system were synchronously recorded during the furnace heating process. The retort was removed from the furnace at the end of the process. The weight-change curve over time was used to determine the degree of weight loss of the material at certain intervals.

Study of the thermal behavior of copper telluride

The reactor was a quartz vessel containing a boat of a specified weight. A detachable porcelain condenser was then placed over the crucible to collect the condensed material. During the experiment, a specified amount of copper telluride was loaded into an alumina crucible. The boat was subsequently placed in the detachable (lengthwise) condenser, which was then inserted into the quartz reactor. A vacuum system was connected to the reactor, and the system was flushed several times with an inert gas (argon). After the gases were evacuated from the system to the specified pressures, the reactor was placed in

the furnace such that the copper telluride load was located in the isothermal zone. In the case of an isothermal process, the furnace was preheated to a predetermined temperature. The start of the experiment was considered to be the moment the set temperature was reached. The reactor was removed from the furnace and cooled under vacuum until the end of the experiment. The resulting residue was then weighed and analyzed. The sample was weighed before and after the experiment using analytical balances PA214C (Ohaus-Pioneer, USA) with an accuracy of ± 0.1 mg.

Characterizations

The material composition was studied by X-ray fluorescence analysis using an Axios 1-kW wavelength-dispersive spectrometer (PANalytical, the Netherlands) with an error of $\pm 5\%$.

X-ray phase analysis was performed using a D8 Advance diffractometer (Bruker, Germany) with Cu-K α radiation. Phase compositions were determined using the ASTM database (PDF-2 reference diffraction database, rel. 2023, International Center for Diffraction Data, USA). For quantitative analysis, the DIFFRAC.EVA software based on the corundum number method was used. The crystalline phase content was quantitatively estimated using the DIFFRAC.EVA program by calculating the ratio of the integrated intensity of the crystalline peaks to the total intensity of the peaks and amorphous background. The amorphous phase content was calculated as the difference between 100% and the crystalline phase content.

Microprobe analysis was performed using a JXA-8230 electron probe analyzer (Jeol, Japan). The electron probe performed under the following conditions: accelerating voltage of 20 kV; beam current of 5 nA.

Results and discussion

Weight-loss mechanism during heating under non-isothermal conditions

The decomposition of the Cu₂Te compound into elemental copper and tellurium does not occur at temperatures below 2704°C (Nitsenko et al. 2023, 2025). However, in an earlier study, we established the possibility of tellurium removal from industrial telluride in an inert atmosphere owing to the presence of oxidizing agents in the material itself (Nitsenko et al. 2022). The total weight loss of the sample was approximately 21%, which was due to the removal of molecular and crystallization moisture as well as tellurium oxide, a decomposition product of the formed copper orthotellurate (Cu₃TeO₆).

According to X-ray phase and X-ray fluorescence analyses, industrial copper telluride, both at the time of delivery (Nitsenko et al. 2020) and the study, contained sufficient amounts of oxygen to extract tellurium in the form of TeO₂.

Previous studies investigated the mechanism of weight loss of industrial copper telluride in an inert atmosphere when heated under non-isothermal conditions at reduced pressure. The experiments were conducted with a temperature increase from 23 to 1100°C, heating rate of 7°C/min, and pressure in the reaction zone of 0.066–91.9 kPa. The dependence of the

weight loss on the continuously increasing temperature at different constant pressures is shown in Figure 4.

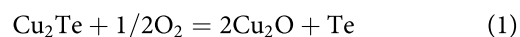
The graph shows that the decrease in the weight of the samples at low pressure occurred with almost identical, non-one-stage characteristics. There are two areas of intensive weight loss (100–350 and 800–1100°C) and one region of relative stability (350–800°C) in all curves, except for the one obtained at atmospheric pressure. Weight change at atmospheric pressure is observed only at temperatures above 800°C. The total weight loss at atmospheric pressure was 21.73%. Lowering the pressure intensifies the extraction of volatile components. Therefore, this figure is 36.2% at a pressure of 0.66 kPa, and 58.7% and 61.2% at 0.133 and 0.066 kPa, respectively.

It can be assumed, based on data obtained for the oxidation – distillation process (Nitsenko et al. 2022, 2022), that the first temperature section on the curves (100–350°C) is associated with moisture loss and the extraction of highly volatile elements and compounds. The absence of this stage at atmospheric pressure is apparently due to the suppression of volatile fraction removal owing to the higher pressure in the system. Phase transformation processes predominate in the range of ‘average’ temperatures (350–800°C). This was accompanied by weight loss at pressures of 0.133 and 0.066 kPa. The final stage is associated with the intensive removal of volatile tellurium dioxide into the vapor – gas phase. Vacuum installations have a high hermeticity; therefore, oxidation of the material due to introduced oxygen was excluded. The transformation of tellurite phases into copper and tellurium oxides occurs under the influence of the oxygen present in the material.

Prediction of phase transformations

When studying the thermal behavior under low-pressure conditions, an important step is to analyze the saturated vapor pressure of elements and compounds capable of transitioning to the vapor – gas phase under the selected process parameters. The presence of elements such as tellurium, oxygen, copper, and chlorine indicates the possibility of forming oxide, chloride, tellurite, and/or tellurate phases during vacuum-thermal treatment, each with characteristic ranges of stability temperature and vapor pressure.

According to Kukleva et al. (1988), with an external oxygen supply to the reaction zone, the oxidation of copper telluride (Cu₂Te) proceeds with the formation of copper(I) oxide and elemental tellurium, which is described by the following reaction:



In the absence of an external oxygen supply, the oxidation of copper telluride is possible because of the oxygen contained in the material, which is released during phase transitions. The first stage is the removal of molecular moisture from the structure of hydrocosulphates at low temperatures, leading to an increase in the amount of brochantite and antlerite phases.

According to Prasad and Sitakara Rao (1985), brochantite (Cu₄(SO₄)(OH)₆), which belongs to the class of basic copper-(II) sulfates, decomposes to form antlerite (Cu₃(SO₄)(OH)₄) in

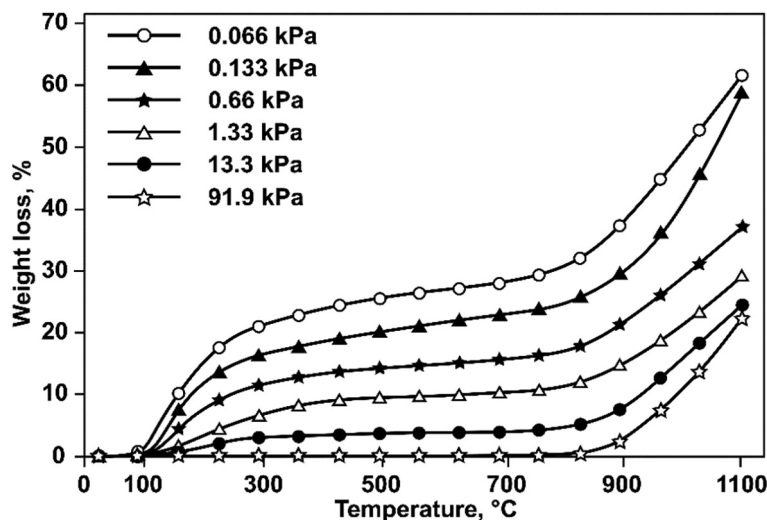
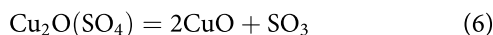
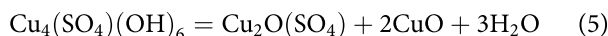
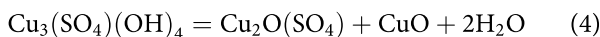
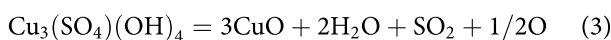
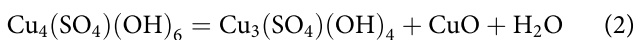


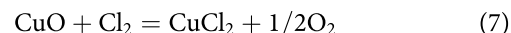
Figure 4. Dependence of copper telluride weight loss on temperature under non-isothermal conditions.

the temperature range of 250–400°C. This stage (Reaction 2) is accompanied by the dehydration and partial dissociation of hydroxyl groups, leading to a decrease in the OH^- content. Upon further heating above 600°C, antlerite, as shown by Koga et al. (2008), undergoes further thermolysis to form CuO, sulfur dioxide (SO_2), residual water, and oxygen (Reaction 3). This reaction represents the final stage of the thermal destruction of copper hydroxosulfates.

It is worth noting that both brochantite and antlerite can transform into an intermediate phase, dolerophanite ($\text{Cu}_2\text{O}(\text{SO}_4)$). These reactions (4, 5) are accompanied by the extraction of water and partial reduction of the sulfate group (Güner et al. 2021; Koga et al. 2008). According to Güner et al. (2021), dolerophanite at temperatures above 600°C decomposes to CuO with the extraction of SO_3 into the gas phase (Reaction 6).



Thus, the thermal behavior of the copper hydroxosulfates is characterized by a multi-stage decomposition mechanism. Particular attention should be paid to the formation of copper(II) oxide, which is the product of all the reactions regardless of the transformation pathway. The formation of CuO plays a key role in subsequent processes, as this oxide can react with chlorine contained in the material. As a result of this interaction, copper chloride (CuCl_2) is formed and oxygen is released (Reaction 7), which can serve as an oxidizer for copper telluride according to Reaction (1). The calculated standard Gibbs energy of Reaction (7) at 500 K is -35.06 kJ/mol, indicating the possibility of the reaction occurring even at moderate temperatures.



In addition to its interaction with copper(II) oxide, chlorine can react with elemental tellurium to form volatile chlorides (Reactions 8 and 9). The calculated standard Gibbs energy of Reaction (8) at 500 K is -179.14 kJ/mol, and that of Reaction 9 is -75.92 kJ/mol.



Among the oxidation reactions, in addition to Reaction (1), the following is expected: elemental tellurium will be oxidized to tellurium oxide (TeO_2), which will then react with copper oxides to form CuTe_2O_5 and/or CuTeO_3 (Kukleva et al. 1988; Zhu et al. 2014) at temperatures of 270–500°C. At approximately 650°C, the formation of the most stable orthotellurate, Cu_3TeO_6 (Dimitriev, Gatev, and Ivanova 1989; Nitsenko et al. 2022; Zhu et al. 2014), should be expected, which, upon further heating, decomposes with the release of tellurium oxide.

A decrease in the system pressure contributes to a shift in the equilibrium of reactions involving gaseous products toward vaporization, leading to a reduction in the temperature at which the substance transitions into the gas phase.

Figure 5 shows the temperature dependence of the vapor pressure of elemental tellurium and tellurium-containing compounds that are most likely formed during the process. As seen in the figure, tellurium tetrachloride is the most volatile tellurium-containing compound. Tellurium dioxide, which is low-volatile up to its melting point (733°C), is the only stable tellurium oxide at temperatures above 430°C (Dimitriev, Gatev, and Ivanova 1989). The saturated vapor pressure of TeO_2 at its melting point of 1006 K (733°C) is 0.026 kPa according to Bolshakov (1976), and approximately 0.019 kPa according to Chizhikov and Shchastliviy (1970). Therefore, the evaporation of TeO_2 should be expected at temperatures above 750°C. It should also be emphasized that the lack of oxygen in the system

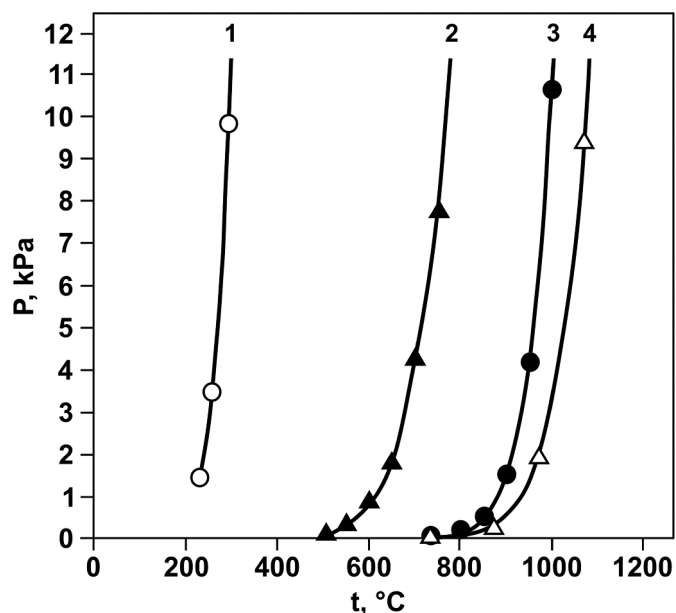


Figure 5. Dependence of saturated vapor pressures of elemental tellurium (2), tetrachloride (1), and tellurium oxides (3, 4) according to Chizhikov and Shchastlivyi (1970).

limits the complete oxidation of tellurium to TeO_2 , which makes the elemental evaporation of tellurium possible.

Phase transformations induced by heating under non-isothermal conditions

The phase compositions of the residues obtained upon heating to 300, 500, 700, 900, and 1100°C and pressures of 0.066 and 13.3 kPa were analyzed to support hypothesis about phase transformation (Figures 6, 7). The heating rate was maintained at 7°C/min. The X-ray phase analysis results demonstrated a consistent trend in the development of phase transitions during the vacuum-thermal and oxidation-distillation processes. The identified phases and their corresponding PDF maps are listed in Table 2.

Thus, the absence of posnjakite ($\text{Cu}_4(\text{SO}_4)(\text{OH})_4$) and copper sulfate hydroxide hydrate ($\text{Cu}_5(\text{SO}_4)_2(\text{OH})_6 \cdot 5 \text{H}_2\text{O}$) phases, which decomposed to brochantite ($\text{Cu}_6(\text{SO}_4)(\text{OH})_6$) and antlerite ($\text{Cu}_3(\text{SO}_4)(\text{OH})_4$) with the release of water molecules into the vapor phase, was observed in the residues corresponding to the first stage of weight loss (up to 350°C). Owing to the high amorphousness of the material (Table 3), only polymorphic transformation was identified for the telluride phases. The residues were well crystallized at 500 and 700°C. The XRD patterns showed pronounced reflections of an unidentified X phase, presumably $\text{Cu}_{0.37}\text{Te}_{0.26}\text{O}_{0.76}\text{S}_{0.084}$, along with copper oxides. Phase X was the predominant phase. Residual traces of copper telluride, antlerite, and dolerophanite ($\text{Cu}_2\text{O}(\text{SO}_4)$, a decomposition product of antlerite) were also observed. X-ray reflections of tellurium oxides, including $\text{Te}_2\text{Cu}_2\text{O}_7$, Cu_3TeO_6 , H_2TeO_4 , and $\text{Cu}_3\text{TeO}_4(\text{OH})_4$, were present as well. The formation of the latter two oxides was likely due to the absorption of hydroxyl groups by tellurium oxide and copper tellurate, resulting from the decomposition of copper hydroxosulfates. Sulfate ions, also formed during the decomposition of copper hydroxosulfates, were removed from

the material into the gas phase, as confirmed by the absence of new sulfur-containing phases in the heat-treated residues.

The processes of the phase transformations are completed at a pressure of 0.066 kPa by a temperature of 900°C. The material contains orthotellurate and copper oxide compounds. In the residue obtained at a pressure of 13.3 kPa, a decrease in the amount of phase X and an increase in the amount of copper(II) oxide were observed. This temperature shift at the onset of the decomposition process of phase X is attributed to the difference between the dissociation pressure of the compound and pressure set by the experimental conditions. The copper orthotellurate phase was observed in very small quantities. The degree of amorphization in both cases increased to 40–44.1%. At 1100°C, only copper(I) oxide was present among the crystalline phases in the residues. The samples exhibited amorphous crystalline structures. The degrees of amorphization of residues obtained at pressures of 13.3 and 0.066 kPa were 47.9% and 29.3%, respectively.

A possible reason for the very small amount of the copper orthotellurate phase at a pressure of 13.3 kPa is the lack of CuO for the reaction $3\text{CuO} + \text{TeO}_2 + 1/2\text{O}_2 \rightarrow \text{Cu}_3\text{TeO}_6$. As shown in Figure 8, the intensity of the copper(II) oxide phase reflexes increases significantly at 13.3 kPa in the temperature range of 500–900°C. This indicates an increase in the amount of this compound. The amount of unidentified phase X decreased slightly, whereas the amount of Cu_2O remained constant. Based on this data, it can be assumed that one of the products of the decomposition of phase X is copper(II) oxide, which, under high temperatures, partially decomposes to copper(I) oxide by the reaction: $2\text{CuO} \rightarrow \text{Cu}_2\text{O} + \text{O}_2\uparrow$. Therefore, the more likely reactions in the range of 900–1100°C, in addition to the reduction of copper(I) oxide, are the formation of copper tellurate phases: $\text{CuO} + \text{TeO}_2 \rightarrow \text{CuTeO}_3$ and $\text{CuO} + 2\text{TeO}_2 \rightarrow \text{CuTe}_2\text{O}_5$, as well as their decomposition. These phases were presumably present in the residue in an amorphous state because of strong disorder in the crystal lattice.

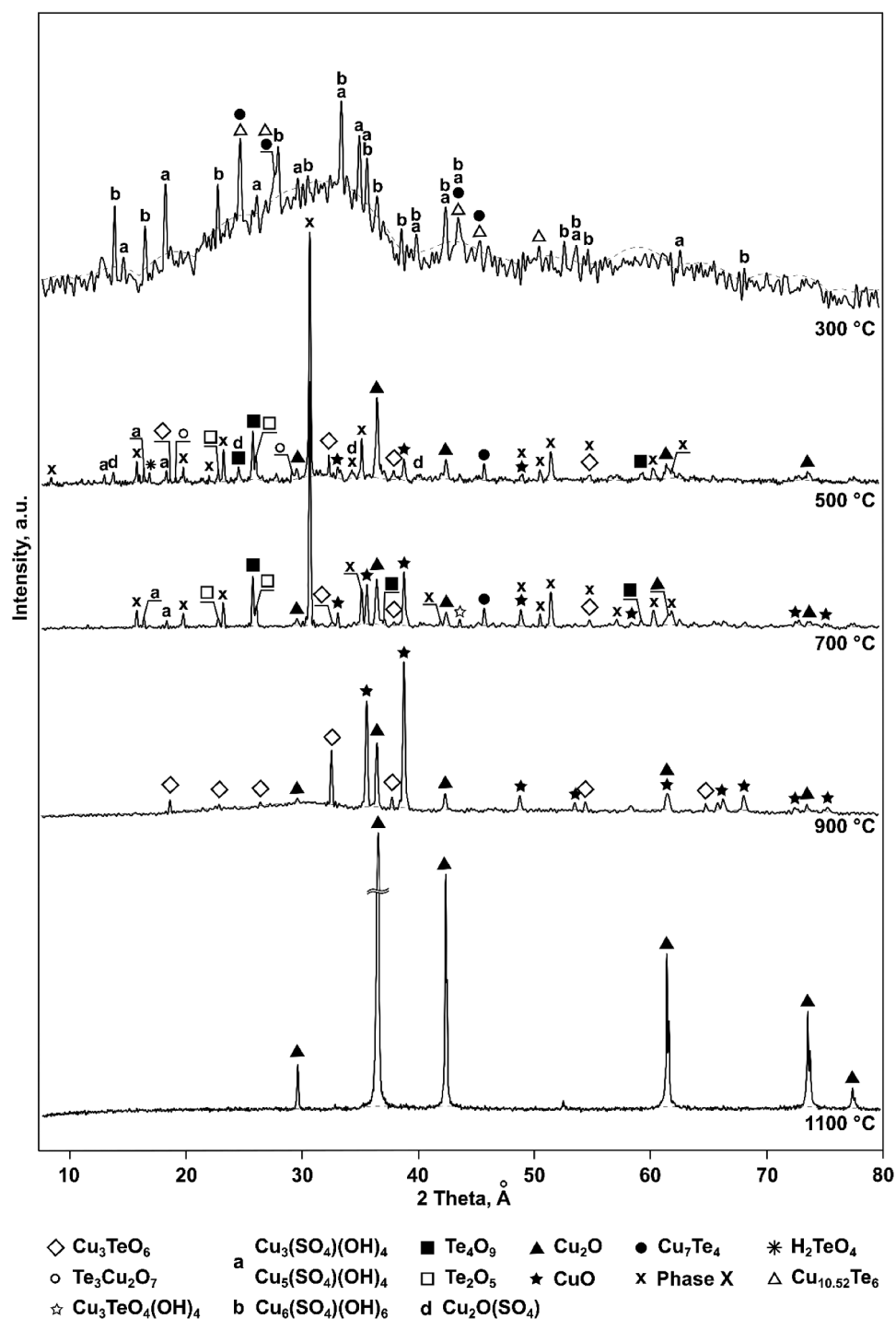


Figure 6. XRD patterns of residues obtained at 0.066 kPa under non-isothermal conditions.

Tellurium recovery

Effect of temperature and pressure on tellurium recovery

Technological tests were conducted to assess the impact of the temperature and pressure during the vacuum-thermal process on the recovery rate of tellurium from copper telluride. The experiments were performed at temperature ranging from 300 to 1100 °C and pressures from 0.066 to 13.3 kPa, with an exposure duration of 1 h. The results are presented in Figures 9, 10. The initial tellurium content in the sample was

0.59 g, with an initial sample mass of 2.5 g. To assess the reproducibility of the experiments, each measurement was performed three times. The resulting data was averaged and used to analyze the effects of the temperature and pressure on the degree of tellurium extraction. Standard deviations ranged from 2% to 2.5%, and standard errors from 1% to 1.4%. These values confirm the acceptable repeatability of the results and consistent behavior of the system under the conditions studied.

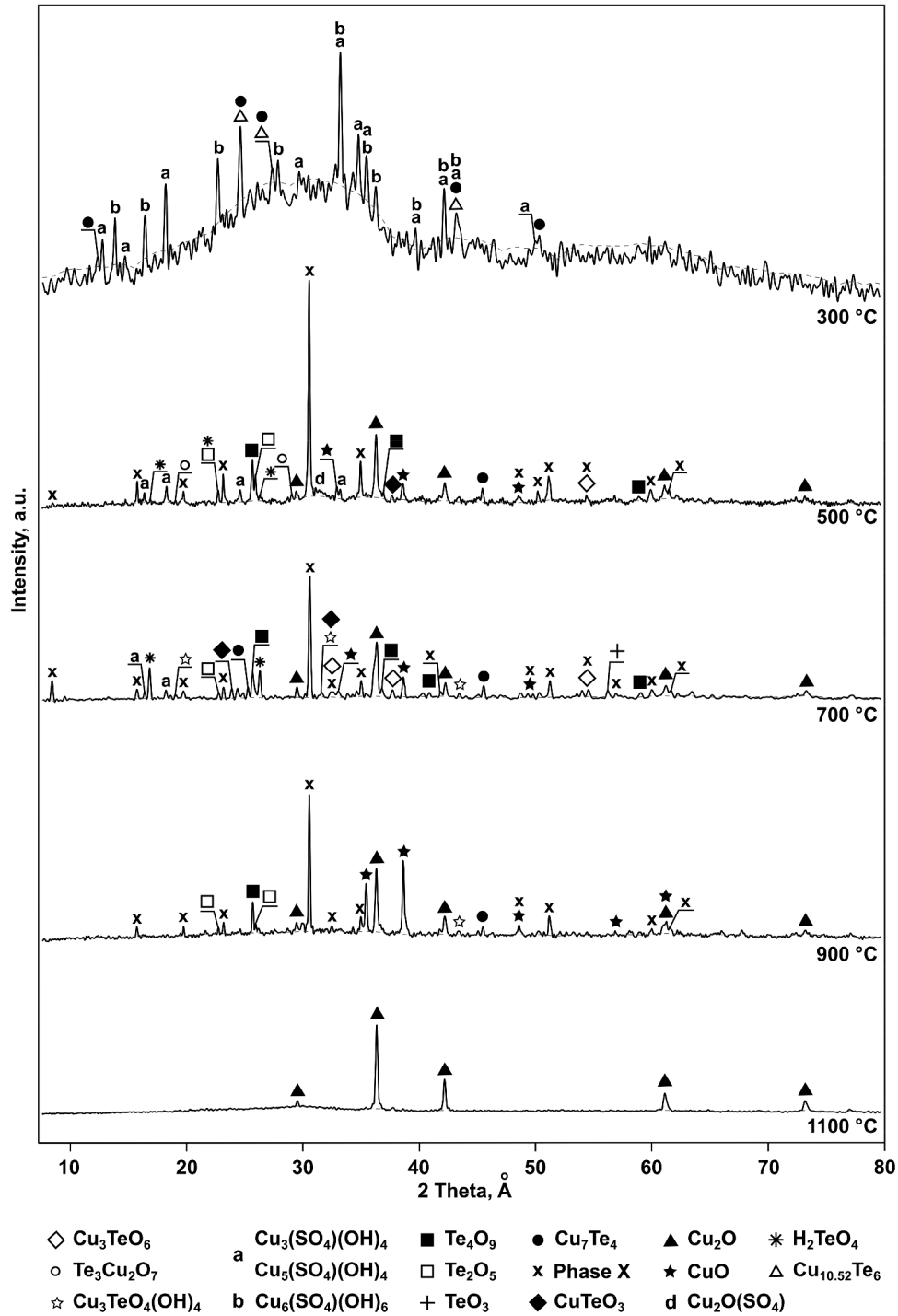


Figure 7. XRD patterns of residues obtained at 13.3 kPa under non-isothermal conditions.

The degree of tellurium extraction (%) was calculated using Equation 10, where m_0 and m_1 are the mass (g) of the initial sample and the resulting residue, respectively; w_0 and w_1 are the tellurium content (%) in the initial sample and the resulting residue, respectively.

$$\frac{m_0 w_0 - m_1 w_1}{m_0 w_0} \times 100 \quad (10)$$

A decrease in the pressure and/or an increase in the temperature is expected to enhance the recovery of tellurium into the condensate. The power curves at all pressures exhibited nonlinear behavior, which corresponds well with the weight-loss curves. As shown in the graph, the highest tellurium recovery rates were achieved at 1100°C and pressures of 0.066 and 0.133 kPa, reaching 98.70% and 96.79%, respectively. The tellurium content in the vacuum residues was

Table 2. Phases identified by X-ray phase analysis.

Phase	Name	PDF	Phase	Name	PDF
Cu ₃ TeO ₆	Copper tellurate	01-070-0701	Cu ₃ (SO ₄)(OH) ₄	Antlerite	01-084-2035
Te ₃ Cu ₂ O ₇	Tellurium copper oxide	00-035-1083	Cu ₅ (SO ₄)(OH) ₄		00-007-0407
Cu ₃ TeO ₄ (OH) ₄	Xocomecatlite	00-029-0579	Cu ₆ (SO ₄)(OH) ₆	Brochantite	00-043-1458
Te ₄ O ₉	Tellurium oxide	01-083-4178	Cu ₂ O(SO ₄)	Dolerophanite	00-013-0189
Te ₂ O ₅	Tellurium oxide	00-025-1113	Cu ₂ O	Copper oxide	01-071-3645
H ₂ TeO ₄	Hydrogen tellurate	01-070-1036	CuO	Tenorite	01-073-6023
Phase X	Not identified		Cu ₇ Te ₄	Copper telluride	00-057-0196
			Cu _{10.52} Te ₆	Copper telluride	01-089-2265

Table 3. Amorphous phase content (%) in residues formed under non-isothermal conditions.

Pressure, kPa	Temperature, °C				
	300	500	700	900	1100
0.067	66.9	31.8	27.7	44.1	29.3
13.3	55.7	34.5	28.5	40.0	47.9

0.72% at 0.066 kPa and 1.62% at 0.133 kPa. Partial tellurium extraction occurred in the range of 300–700°C at pressures below 1.33 kPa, accompanied by phase transformation processes. However, a noticeable effect is exerted by the decomposition of copper orthotellurate at temperatures above 700°C and the attainment of the evaporation temperatures of TeO₂. At 900°C, the evaporation rate decreases due to the depletion of the material in tellurium. At a pressure of 13.3 kPa, the shape of the curve differs from those obtained at pressures below 1.33 kPa. A sharp increase in the degree of tellurium evaporation is observed at temperatures above 900°C, which is associated with the attainment of conditions close to the evaporation temperature of TeO₂ at a pressure of 13.3 kPa.

The tellurium extraction curves from industrial copper telluride were analyzed to determine the kinetic parameters. The obtained data were processed using the dimensionless differential kinetic equation proposed by Wendlandt (1974), which is expressed as:

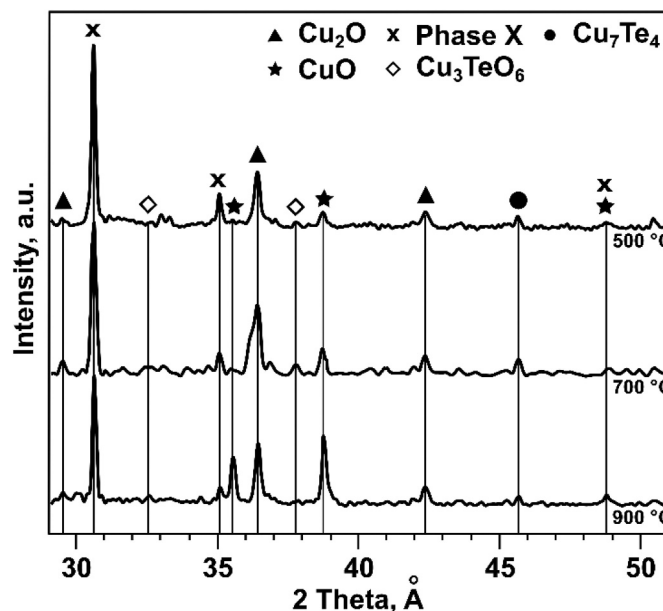
$$d\alpha/d\tau = k(1 - \alpha)^n \exp(-E/RT) \quad (11)$$

where $d\alpha/d\tau$ (denoted as V) represents the rate of the process, k is the reaction rate constant, α is the fraction of the reacted substance, n is the reaction order, E is the apparent activation energy, and R is the universal gas constant.

Dividing the left side of Equation (11) by $(1 - \alpha)^n$ and taking the logarithm of the resulting expression yields:

$$\lg[(d\alpha/d\tau)(1 - \alpha)^n] = -E/RT + \ln A = -A/T + B \quad (12)$$

The value under the logarithmic sign on the left-hand side of Equation (12) represents the reduced dimensionless rate. The apparent activation energy was determined from the constant A , and the equilibrium constant was determined from the constant B . The reaction order was assumed to be equal to one, based on the following reasoning: the reaction rate during the evaporation of volatile components is primarily governed by the conditions of the heat supply and diffusion of these

**Figure 8.** Fragments of XRD patterns of residues obtained at 500, 700, and 900°C and a pressure of 13.3 kPa (from Figure 7).

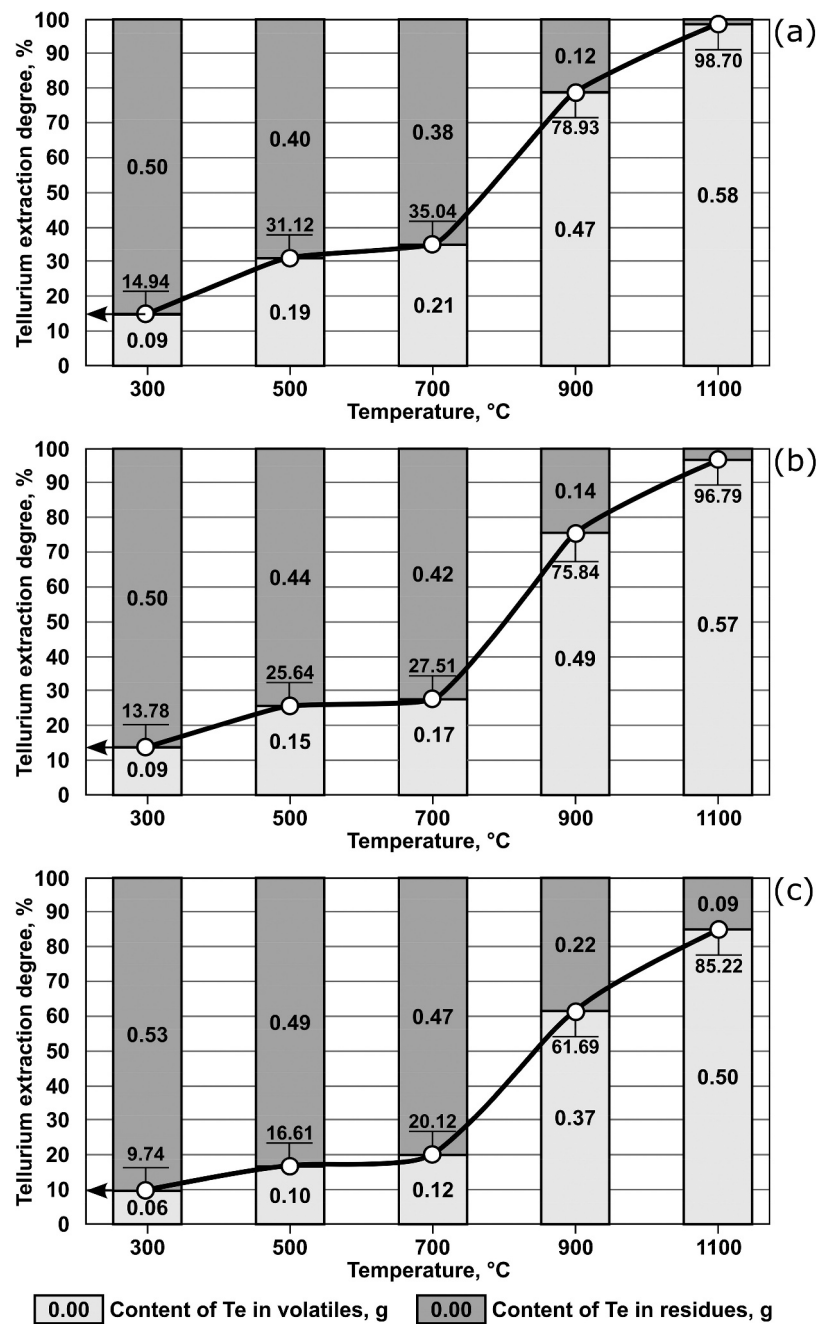


Figure 9. Dependence of the degree of tellurium evaporation on constant temperature and tellurium content in products of vacuum heat treatment at pressures of 0.066 (a), 0.133 (b), and 0.667 kPa (c).

components through the layer. In this case, the area of the heat supply changes in direct proportion to the remaining mass, and the growth of the inert component layer adversely affects both the diffusion process and heat supply through the resulting layer. Therefore, the most probable value of the order of the thermal decomposition reaction at reduced pressure should be less than or approximately equal to the first value.

Table 4 presents the results of the kinetic parameter calculations for tellurium extraction, while Figure 11 illustrates the results of the experimental data processing.

As shown in Figure 11, the dependences of $\lg[(da/d\tau)(1-\alpha)^n] - 1/T$ at pressures of 0.066 to 1.33 kPa in the temperature

range of 300 to 1100°C are nonlinear, with an inflection point occurring at 700°C. This indicates that the process proceeds in two areas with different limiting factors. When the dependence is divided into two segments (300–700 and 700–1000°C), straight lines are obtained, showing that the process follows the Arrhenius equation. The correlation coefficients (R^2) range from 0.92 to 1, supporting the applicability of the obtained equation to describe the experimental data.

According to the formal interpretation, it can be concluded that during 300–700°C, the process occurs in the diffusion region and is characterized by the formation and release of a volatile tellurium-containing compound. In this case, the

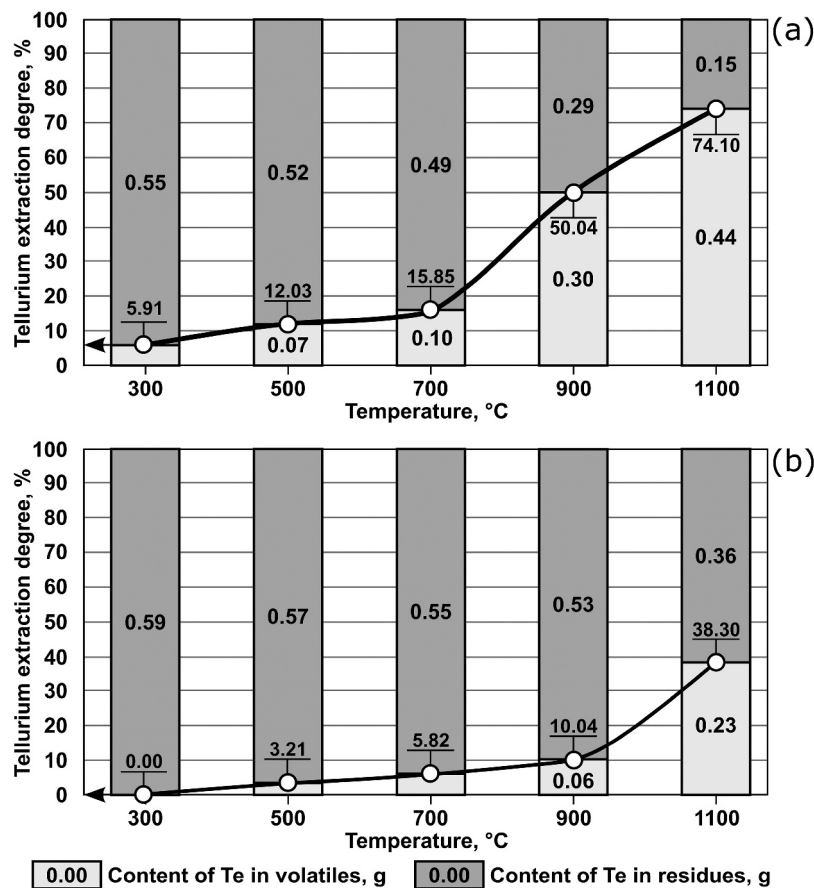


Figure 10. Dependence of the degree of tellurium evaporation on constant temperature and tellurium content in products of vacuum heat treatment at pressures of 1.33 (a) and 13.3 kPa (b).

Table 4. Kinetic parameters of tellurium extraction under isobaric – isothermal conditions and approximation reliability values (R^2).

Pressure, kPa	Kinetic parameters in the temperature ranges of									
	Coefficients of the equation $\lg[da/d\tau/(1-a)^n]$ $= -A/T + B$					Coefficients of the equation $\lg[da/d\tau/(1-a)^n]$ $= -A/T + B$				
	A	B	E_{apparent} , kJ/mol	k , s^{-1}	R^2	A	B	E_{apparent} , kJ/mol	k , s^{-1}	R^2
	(573–973 K) 300–700 °C					(973–137 K) 700–1100 °C				
0.066	–1321.3	–2.72	10.98	0.8	0.94	–10834	4.76	90.03	32755.0	0.95
0.133	–1025.9	–3.11	8.52	0.3	0.92	–9666.7	3.73	80.33	6383.0	0.97
0.66	–985.08	–3.35	8.19	0.2	0.99	–6985.9	1.46	58.05	162.87	0.99
1.33	–1274.2	–.24	10.59	0.4	0.99	–6082.9	0.62	50.55	43.73	0.99
	(573–1173 K) 500–900 °C					(1173–1373 K) 900–1100 °C				
13.3	–1980.4	–3.15	16.46	0.2	0.99	–8869.3	1.63	73.70	30.53	1

displayed response energy does not depend on the pressure. However, a decrease in pressure minimizes the resistance to the external environment and prevents the removal of molecular compounds from the surface of the material grains. The observed slowdown in the rate of tellurium evaporation between 500 and 700 °C is apparently due to a reduction in the amount of volatile compound on the grain surface and oxidation of tellurium to form nonvolatile copper tellurates and tellurites. In the temperature range of 700–1100 °C, the process apparently occurs in the kinetic-diffusion region and is

governed by both the energy required to break the CuO – TeO₂ molecular bonds in the previously formed compounds and transfer of matter to the vapor – gas phase. In this case, the influence of the indicated diffusion factor on the overall process is expected to decrease as the pressure decreases, leading to an increase in the apparent activation energy and rate constant. It is likely that at pressures of 0.066 and 0.133 kPa and temperatures above 900 °C, tellurium evaporation is also influenced by intracrystalline diffusion, caused by the depletion of TeO₂ molecules on the solid surface. At a pressure of

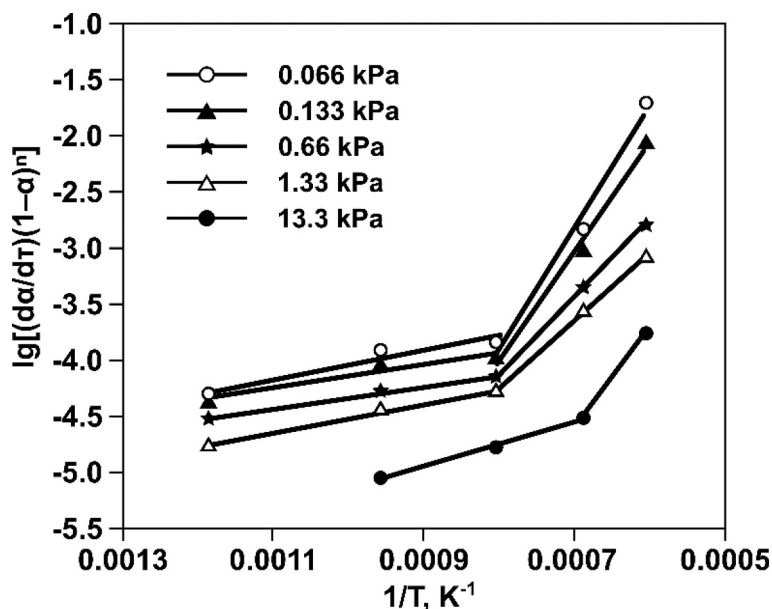


Figure 11. Dependence of the reduced evaporation rate of tellurium on temperature for a first-order reaction ($n = 1$).

13.3 kPa, a deviation in the dependence of $\lg[(d\alpha/d\tau)(1-\alpha)^n]$ versus $1/T$ is observed at 900°C. Under these conditions ($t = 500\text{--}1100^\circ\text{C}$, $p = 13.3\text{ kPa}$), external diffusion resistance plays a dominant role, and evaporation is limited by mass transport in the gas phase.

Analysis of vacuum residues

According to the X-ray phase analysis data, in the temperature range from 300 to 700°C, the phase composition of the resulting residues differs little from that presented in Figures 6, 7. Differences were observed for samples obtained at 900°C, which consisted of crystalline copper oxides in varying ratios and amorphous components at all specified pressures (Figure 12). Electron probe microanalysis of the residues obtained at pressures of 0.066 and 13.3 kPa was performed to provide more comprehensive information about the phase composition (Figure 13).

It was determined that the tellurium in the material was concentrated in a compound similar in composition to CuTeO_3 , which was not detected by X-ray phase analysis. Copper metatellurate served as a matrix cementing the copper oxide grains. The copper(II) oxide consisted of grains of various shapes and sizes with well-defined boundaries. Copper(I) oxide forms marginal edges along the periphery of CuO grains or completely replaces them. Impurities contained in the source material that did not sublime during vacuum heat treatment appeared as spherical and oval agglomerates at the interface between the oxide and copper tellurium phases. This alloy morphology resulted from the sequential crystallization of compounds, leading to a solid solution decay structure during cooling.

The results obtained at 0.066 kPa suggest that the decomposition of copper orthotellurate to copper(I) oxide occurred in three successive steps. The first stage is characterized by the formation of two main phases of copper metatellurate and copper(II) oxide in the melt, accompanied by the evaporation of tellurium oxide according to the reaction: $2\text{Cu}_3\text{TeO}_6 \rightarrow \text{CuTeO}_3 + 5\text{CuO} + \text{TeO}_2\uparrow + \text{O}_2\uparrow$. The formed CuO grains, in a sense, serve as nuclei for nucleation, so the second stage begins at their boundaries,

which is the decomposition of copper metatellurate with the release of additional copper(II) oxide and tellurium oxide: $\text{CuTeO}_3 \rightarrow \text{CuO} + \text{TeO}_2\uparrow$. The third stage involves the reduction of copper(II) oxide to copper(I) oxide: $2\text{CuO} \rightarrow \text{Cu}_2\text{O} + 1/2\text{O}_2$.

It is possible to outline the following scheme for the processes occurring in the material at a pressure of 13.3 kPa and temperature of 900°C. The stage of active decomposition of phase X begins at 900°C. Its products include copper(II) oxide and tellurium oxide. One part of the formed oxide was reduced to Cu_2O , while the second part reacted with tellurium oxide to form CuTeO_3 and a very small amount of Cu_3TeO_6 . The saturated vapor pressure of tellurium oxide at this temperature was approximately 1.33 kPa (Bolshakov 1976). Therefore, the extraction of tellurium (Figure 10b) is not significant. Metatellurate breaks down into tellurium and copper oxides with a further increase in temperature. It was not possible to determine the probability of its degradation at 900°C based on the available data; further studies are required.

Conclusion

The comprehensive studies carried out in this work have, for the first time, demonstrated the possibility of substantially lowering the temperature of the pyrometallurgical process by extracting tellurium in oxide form under low-pressure conditions in an inert atmosphere. The results obtained indicate the following:

- (1) The conducted theoretical analysis and the obtained X-ray phase analysis data showed that the formation of tellurium oxide occurred due to oxidation processes in the material containing oxygen in its composition. Except for tellurium oxide, evaporation of tellurium in elemental form and in the form of tetrachloride is assumed. It was found that at 0.066 kPa, industrial copper telluride was oxidized through intermediate compounds to Cu_3TeO_6 , which at 900°C decomposed

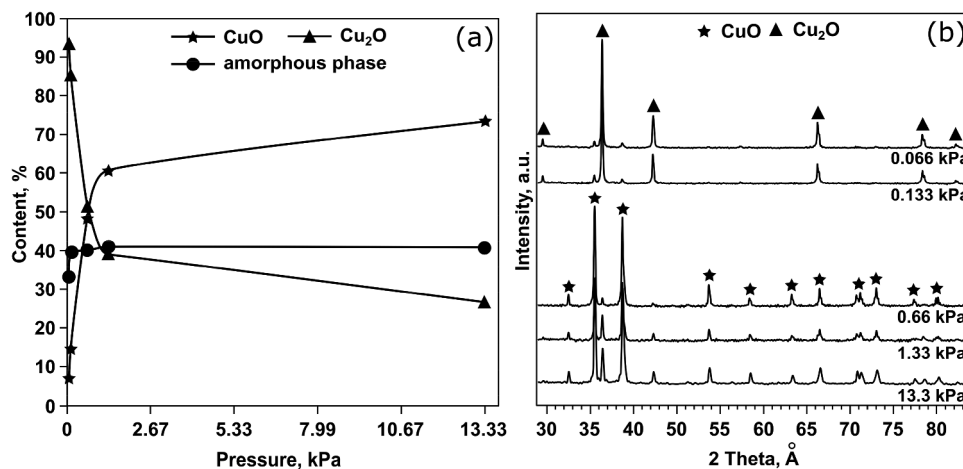


Figure 12. Content of copper oxides in residues and their amorphousness (a), and XRD patterns of residues (b).

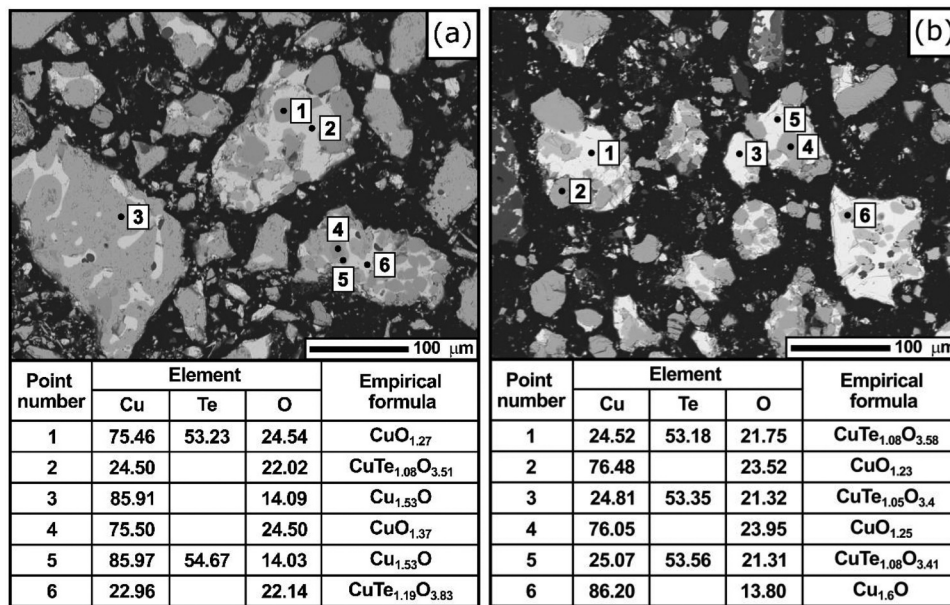


Figure 13. SEM results of residues obtained at 900°C and pressures of 0.066 (a) and 13.3 kPa (b).

through CuTeO₃ to CuO and TeO₂. The process proceeded without the formation of the Cu₃TeO₆ phase at a pressure of 13.3 kPa.

- (2) Technological tests have shown that a decrease in pressure and/or increase in temperature positively affected the degree of tellurium recovery into the condensate. The best results were obtained at a temperature of 1100°C and pressures of 0.066 and 0.133 kPa (98.70% and 96.79%, respectively). The tellurium content in the vacuum residues was 0.72% at 0.066 kPa and 1.62% at 0.133 kPa.
- (3) Kinetic analysis of the tellurium evaporation process indicated that during 300–700°C, the process occurred in the diffusion region and was characterized by the formation and presence of a volatile tellurium-containing compound. In the temperature range of 700–1100°C, the

process apparently occurred in the kinetic-diffusion region and was determined both by the energy required to break the CuO – TeO₂ molecular bonds in the previously formed compounds and transfer of matter to the vapor – gas phase. In this case, the influence of the indicated diffusion factor on the overall process was expected to decrease as the pressure decreased, leading to an increase in both the apparent activation energy and rate constant.

The resulting copper residue in the form of oxides (CuO and Cu₂O) was sent for further processing to obtain metallic copper. Prometallurgical methods were used for this purpose, including the reduction of oxides with carbon-containing reducing agents at elevated temperatures, which converted copper to its metallic state. Alternatively, hydrometallurgical processes based on acid leaching, followed by the

electrochemical or chemical recovery of copper from the solution, were employed. Thus, recycling oxide residues ensured the return of copper to the production cycle and contributed to increasing the complexity of raw material utilization.

The next step in the research is to determine the optimal process parameters and conduct larger-scale tests.

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Disclosure statement

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