PGM synthesis and CO_2 adsorption

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1 Introduction

Pure minerals for research purposes are not available in nature. Mineral synthesis offer the way to investigate pure mineral surface properties. The use of greenhouse gas CO_2 as a reagent on sulphide-poor PGM (Platinum Group Minerals) concentration has been under investigations lately. This paper presents PGM synthesis test work and CO_2 adsorption results on some synthetic PGM.

2 Mineral synthesis and CO_2 adsorption tests

2.1 Mineral synthesis procedure

For CO_2 adsorption tests, $PtAs_2$, PdTe, $PdTe_2$, $PtTe_2$ and $NiTe_2$ were synthetized by using a modified high-temperature process described in Shackleton et al. 2007a and in Shackleton et al. 2007b. As a preliminary step, a set of quartz tubes with two different diameters (6/4 mm and 10/8 mm) and one end sealed were prepared. The tubes were cut to suitable length and sealed with air-acetylene torch. They were then washed with aqua regia and distilled water to remove any traces of contamination and dried. Raw materials in synthetic minerals production are listed in Table 1.

Table 1 *Raw materials of the mineral synthesis.*

Reagent	Specifications
Platinum	Alfa Aesar 012100, 99.9%, <10 µm powder
Palladium	Alfa Aesar 010961, 00.99+%, 0.5mm wire
Arsenic	Goodfellow AS006125, 99.99%, lump
Tellurium	Goodfellow TE006105, 99.95%, lump
Nickel	Alfa Aesar 044739, 99.8 % 150-200 mesh powder

If the reagent was not in a powder form it was first grinded or, in the case of wire, cut to small pieces. Stoichiometric amounts of reagents were weighted and inserted into the 6/4 mm tube. The tube was then connected to a vacuum-gas line system. A schematic picture of the mineral synthesis set-up is presented in Figure 1.

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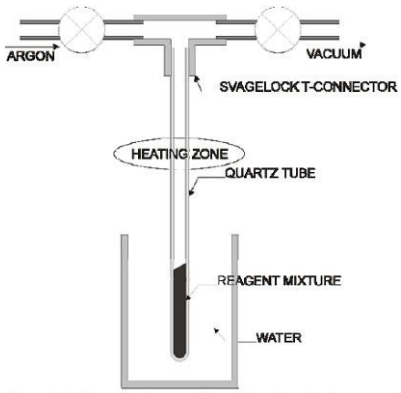


Figure 1 *Schematic picture of the mineral synthesis preparation system.*

During one vacuum-gas insertion cycle the tube was first evacuated to pressure of 10 mbar or less and then filled with 5.0 grade argon gas up to 2 bar. The cycle was repeated ten times, and finally, the tube was evacuated to 10 mbar or less. The lower end of the tube was immersed in water and then the tube was sealed by using air-acetylene torch and the excess of tubing was cut off. For safety reasons the 6/4 mm tube was finally sealed to a 10/8 diameter tube by using the vacuum-gas insertion cycle described above. Sealed tubes were then heat-treated to form the required compounds. For temperatures and reaction times, see Table 2. Cooled reagents were then removed from the reaction tubes and characterized by SEM-analysis.

Table 2 *Preparation conditions of the minerals.*

Compound	Temperature (°C)	Reaction time (min)
$PtAs_2$	800	360
$PtTe_2$	800	60
PdTe	1150	15
$PdTe_2$	1150	15
$NiTe_2$	1150	15

CO_2 adsorption tests

CO_2 adsorption on mineral surfaces was measured by using Agilent 6890–Perkin Elmer ATD-650 GC/MS-ATD equipment. The standard tubes used in thermal desorption analysis were filled with synthesized mineral samples that were powdered with a mortar. CO_2 was then passed through the tube at the rate of 1 l/min at room temperature. Used CO_2 was supplied by AGA Finland and its purity was 99.7% CO_2. Sample tubes were analyzed after CO_2 treatment to find out the amounts of desorbing CO_2 when heating the mineral up to temperature 250 °C.

The thermal desorption method of the sample tubes used here was a two-stage desorption method with three minutes primary desorption time and one minute secondary desorption. At primary desorption the sample tube was heated to 250 °C and held at that temperature for three minutes. The adsorbed CO_2 was desorbed and carried with helium to the cold trap where the temperature was – 30 °C. At the secondary desorption stage the cold trap was heated rapidly (in 6 seconds) to 250 °C and kept at that temperature for one minute. From the cold trap the desorbed CO_2 continued to GC column in He flow. The purpose of the fast heating of the cold trap is to have a similar injection to the GC column compared to the liquid sample injection.

3 Results

3.1 Analysis of the synthetic minerals

For SEM analysis the synthesized mineral nuggets were mounted on small aluminum mounts with double-sided adhesive carbon tabs and conductive carbon cement. Then they were carbon-coated to ensure conductivity all around the samples and then analyzed with a Zeiss Ultra Plus scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS) for elementary analysis. The analytical conditions used for this study were as follows: Acceleration voltage 15kV, beam current 2.3 nA, and a live counting time of 60 seconds for spectra. The standards used for nickel, palladium and platinum were pure metals whereas synthetic InAs and HgTe standards were used for arsenic and tellurium, respectively. A SEM picture of the synthetic kotulskite is presented in Figure 2. The elementary analysis spectrum and non-normalized analysis result are shown in Figure 3.

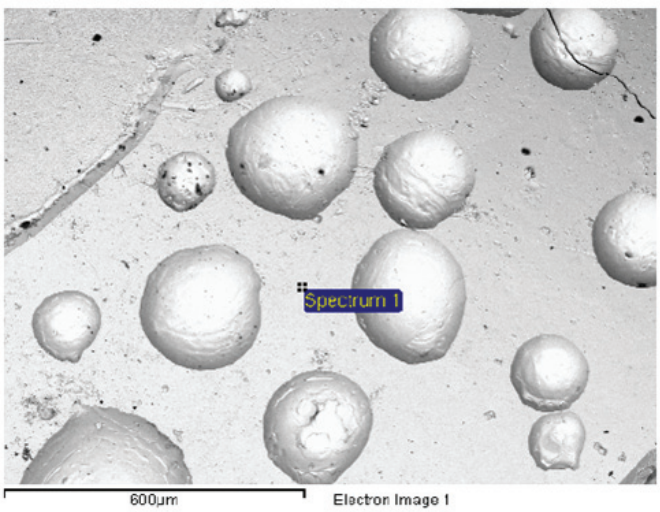
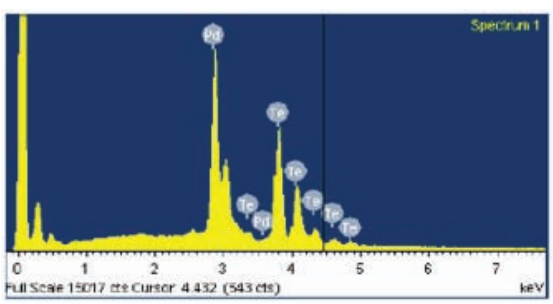


Figure 2 *The SEM picture of the synthetic kotulskite.*



Element	Weight%	Atomic%
Pd L	44.56	50.43
Te L	52.53	49.57
Totals	97.09	

Figure 3 *The EDS spectrum and non-normalized analysis result of the synthetic kotulskite.*

3.2 CO_2 adsorption test results

CO_2 adsorption test results are presented in Table 3. CO_2 adsorption occurred on $PtAs_2$, PdTe, $PdTe_2$ and $PtTe_2$ but no on $NiTe_2$.

Table 3.

	$PtAs_2$	$PtTe_2$	PdTe	$PdTe_2$	$PdTe_2$
CO_2 adsorption	X	X	X	X	
NO adsorption					X

4 Future work

In future, synthetic minerals will be used in flotation behaviour studies. Flotation behaviour of the synthetic mineral will be estimated by zeta-potential measurements and microflotation tests.

References

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