

## NEW OCCURRENCES OF HESSITE, PETZITE AND STÜTZITE AT CORANDA-HONDOL OPEN PIT (CERTEJ GOLD-SILVER DEPOSIT, ROMANIA)

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**Abstract:** Au-Ag- and Ag-tellurides are widely abundant in the vast majority of the “Golden Quadrilateral” (Metaliferi Mts., Romania) ore deposits. Optical microscope observations, electron microprobe and  $\mu$ -Raman analyses have been successfully carried out to identify Au-Ag-Te minerals from Coranda-Hondol open pit (part of Certej deposit, South Apuseni Mountains, in Romania). The identified tellurides are hessite ( $\text{Ag}_2\text{Te}$ ), petzite ( $\text{Ag}_3\text{AuTe}_2$ ) and stützite ( $\text{Ag}_{5-x}\text{Te}_3$ , where  $x = 0.24-0.36$ ) and they usually occur as patches (reaching 90-100  $\mu\text{m}$  in length) in galena, bournonite-seligmannite or at the contact between the last three minerals. In general, all the microanalytical data are stoichiometric with a slightly compositional variation in the case of hessite. The  $\mu$ -Raman spectrum of stützite shows a characteristic Raman fingerprint with a very strong band at 147  $\text{cm}^{-1}$ . The broadness of the Raman band (147  $\text{cm}^{-1}$ ) indicates a non-homogeneous distribution and, in conjunction with the strong heterogeneity observed under microscope, it suggests that the tellurides have formed contemporaneously and they can possibly be attributed to a later silver-rich telluride-bearing substage.

**Keywords:** Certej, Coranda-Hondol, tellurides, hessite, petzite, stützite, EPMA, Raman spectroscopy

### 1. INTRODUCTION

The “Golden Quadrilateral” (Metaliferi Mts., Romania) is well-known for the telluride-bearing epithermal ore deposits (Ciobanu et al., 2004b; Cook & Ciobanu, 2004; Cook et al., 2004).

Besides the economic interest of Au-Ag tellurides they bare, they are also a good indicator of gold (also known as gold carrier) (Ciobanu et al., 2004b; Tamas et al., 2006; Maslennikov et al., 2012).

By far, the most important deposit of Au-Ag- and Ag-tellurides from the “Golden Quadrilateral” is the Săcăriș ore deposit, where the Au/Te ratio is 1:2 (Udubașa & Udubașa, 2004). Also, many tellurides have been described for the first time in the world at the Săcăriș ore deposit (e.g. nagyágite, petzite, stützite, museumite, etc.) (Bindi & Cipriani, 2004; Ciobanu et al., 2004a).

The Au-Ag- and Ag-tellurides are generally

less abundant in other deposits from the “Golden Quadrilateral”, i.g. Brad (Musariu), Fața Băii – Larga (Horia level, 516 m), Baia de Arieș, etc. (Cook et al., 2004).

To the best of our knowledge, only one telluride, i.e. kostovite ( $\text{CuAuTe}_4$ ) was mentioned at the Coranda-Hondol open pit, but its presence was not confirmed by any analytical technique (Udubasa et al., 1992).

The current study presents new occurrences and compositional data on the Au-Ag- and Ag-tellurides from Coranda-Hondol open pit. The hessite ( $\text{Ag}_2\text{Te}$ ), petzite ( $\text{Ag}_3\text{AuTe}_2$ ) and stützite ( $\text{Ag}_{5-x}\text{Te}_3$ , where  $x = 0.24-0.36$ ) were identified by means of EPMA (Electron Probe Microanalyzer) and Raman spectroscopy techniques. Also, the presence of these tellurides was optically and texturally characterized. Furthermore, the temperature conditions regarding the formation of

these ternary assemblages hessite-petzite-stützite are taken into discussion.

## 2. GEOLOGY

The Coranda-Hondol open pit (CHOP) is located at the southern extremity of the “Golden Quadrilateral”, South Apuseni Mountains (45° 59' 37" N, 23° 00' 27" E, Metaliferi Mts., Romania). This area was mined for gold and silver (late 17<sup>th</sup> century) and it recently hosted operations focused mainly on the extraction of Pb and Zn. The ore from CHOP is part of the Certej metalliferous ore deposit.

The main metalliferous minerals from the CHOP are galena (PbS), Fe-poor sphalerite (Zn,Fe)S (1-3% Fe, Udubaşa et al., (1982) and pyrite (Fe<sub>2</sub>S). Chalcopyrite (CuFeS<sub>2</sub>) and sulfosalts occur only at microscopic level. Chalcopyrite mainly occurs as “chalcopyrite disease” in sphalerite. The gangue minerals (of no economic value) are quartz, calcite, rhodocrosite, barite and adularia (potassium aluminosilicate).

Several reviews have described the geological setting and geology of the Coranda-Hondol open pit (Ianovici et al., 1976; Udubaşa et al., 1982; Alderton et al., 1998; Pricopie et al., 2004; Ageneau et al., 2006; Forward et al., 2009; Apopei et al., 2012).

## 3. MATERIALS AND METHODS

The samples were randomly collected from the entire perimeter of the Coranda-Hondol open pit. The telluride minerals usually occur as patches which can reach 100 µm in size and only grains larger than 10-20 µm were could be observed at a magnification up to 20X. Most probably there are other grains which contain one or more telluride minerals, but they are observable only at high magnification as it is the case of point 6 (hessite, observed during EPMA analyses) from figure 1g.

The identity of the telluride minerals has been established firstly based on their optical properties, and then it was confirmed by EPMA and in the case of stützite by micro Raman measurement.

An optical microscope MEIJI ML9430 was used for the optical observations in reflected light (air technique), having a DSLR camera (Canon<sup>®</sup> 450D) attached to it for the microphotographs.

The mineral phases were analyzed on a Cameca SX-100 Electron Probe Microanalyzer (EPMA), at the State Geological Institute of Dionyz Stur (Bratislava), equipped with one ED (energy-dispersive) and four WD (wavelength-dispersive) spectrometers. All the measurements were performed on carbon coated polished sections using

an acceleration voltage of 25 kV, 4 µm beam diameter and the following lines and standards: S K $\alpha$  (CuFeS<sub>2</sub>), Fe K $\alpha$  (CuFeS<sub>2</sub>), Pb M $\alpha$  (PbS), Cu K $\alpha$  (Cu), Ag L $\alpha$  (Ag), As K $\alpha$ , L $\alpha$  (GaAs), Sb L $\beta$  (Sb), Se L $\beta$  (Bi<sub>2</sub>Se<sub>3</sub>), Te L $\alpha$  (Bi<sub>2</sub>Te<sub>3</sub>), Hg L $\alpha$  (HgS), Bi L $\alpha$  (Bi), Zn K $\alpha$  (ZnS), Mn K $\alpha$  (Mn), Au L $\alpha$  (Au).

The non-polarized micro-Raman spectrum was obtained using a Horiba - Jobin Yvon - Labram spectrometer, equipped with an Olympus microscope. The 632.81 nm line of a He-Ne laser was used as excitation, 600 lines/mm holographic grating and CCD detector were used. The instrument includes confocal mode system. The laser power was controlled by means of a series of density filters in order to avoid heating effects.

## 4. RESULTS AND DISCUSSIONS

### 4.1. Optical and textural features

The polished section (sample number CH06/3A) was studied firstly by using an optical microscope in reflected light (air technique) at magnification up to 20X. In plane-polarized incident light the hessite (Ag<sub>2</sub>Te) is grey with slightly brownish tint, scratches, while in crossed nicols it shows a dark orange and bluish distinct anisotropy (see Fig. 1b, 1c, 1c1 and 1h). At this magnification of the microscope (20X) the optical differences between hessite and petzite (Ag<sub>3</sub>AuTe<sub>2</sub>) are unnoticeable (Cabri, 1965; Pracejus, 2008).

Ag- and/or Au-Ag-tellurides usually occur as patches (Fig. 1d, 50-100 µm in size) in galena (Gn), in bournonite-seligmanite (Bnn-Slg) (Fig. 1g, 10 µm in size) and at the contact between galena and bournonite-seligmanite (Figure 1c and 1c1, 80 µm in size). Ag- and/or Au-Ag-tellurides can also be observed inside sulfosalts (bournonite-seligmanite) veins (Fig. 1h, 5-10 µm in width and 30 µm long).

### 4.2. Electron microprobe analyses

Electron microprobe analyses reveal the presence of hessite and petzite. Based on the results of the chemical composition (Table 1), the content of Ag and Te in hessite is from 57.35 wt.% to 60.61 wt.% and from 35.77 wt.% to 38.35 wt.%, respectively. For petzite, the content of Ag, Te and Au is 41.90 wt.% to 42.07 wt.%, 29.88 wt.% to 32.31 wt.% and 22.74 wt.% to 23.91 wt.%, respectively. The empirical formulas calculated on the basis of the total number of atoms are shown in table 1. The microanalytical data give the empirical mean composition Ag<sub>3.06</sub>Au<sub>0.93</sub>(Te<sub>1.92</sub>Se<sub>0.05</sub>S<sub>0.01</sub>) $\Sigma=1.98$  for petzite, and Ag<sub>1.96</sub>Te<sub>1.02</sub> for hessite.

Table 1. Electron microprobe analyses of Au-Ag tellurides from the Certej deposit (Coranda-Hondol open pit) (wt.%).

| sample point # | Analysis (wt.%) |    |       |    |      |    |    |      |       |      |      | Mineral |       |  |         |
|----------------|-----------------|----|-------|----|------|----|----|------|-------|------|------|---------|-------|--|---------|
|                | S               | Pb | Ag    | Cu | As   | Sb | Fe | Se   | Te    | Hg   | Bi   |         | Au    | Total  |         |
| p1             | 0.03            | 0  | 60.61 | 0  | 0.21 | 0  | 0  | 0    | 36.43 | 0.02 | 0    | 0.04    | 97.33 | (Ag <sub>1.98</sub> As <sub>0.01</sub> ) <sub>Σ=1.99</sub> Te <sub>1.01</sub>                                      | Hessite |
| p2             | 0.02            | 0  | 60.24 | 0  | 0.06 | 0  | 0  | 0.06 | 35.95 | 0.06 | 0.04 | 0.03    | 96.47 | Ag <sub>1.99</sub> Te <sub>1.00</sub>  | Hessite |
| p3             | 0.05            | 0  | 60.59 | 0  | 0.07 | 0  | 0  | 0.08 | 35.77 | 0    | 0.03 | 0.05    | 96.64 | Ag <sub>1.99</sub> (Te <sub>0.99</sub> S <sub>0.01</sub> ) <sub>Σ=1.00</sub>                                       | Hessite |
| p4             | 0.09            | 0  | 42.07 | 0  | 0.02 | 0  | 0  | 0.61 | 29.88 | 0.01 | 0    | 23.91   | 96.59 | Ag <sub>3.09</sub> Au <sub>0.96</sub> (Te <sub>1.86</sub> Se <sub>0.06</sub> S <sub>0.02</sub> ) <sub>Σ=1.94</sub> | Petzite |
| p5             | 0.07            | 0  | 41.90 | 0  | 0.12 | 0  | 0  | 0.53 | 32.31 | 0.04 | 0    | 22.74   | 97.71 | Ag <sub>3.04</sub> Au <sub>0.90</sub> (Te <sub>1.98</sub> Se <sub>0.05</sub> ) <sub>Σ=2.03</sub>                   | Petzite |
| p6             | 0.12            | 0  | 57.35 | 0  | 0.10 | 0  | 0  | 0.03 | 38.35 | 0.01 | 0    | 0.09    | 96.05 | Ag <sub>1.90</sub> (Te <sub>1.08</sub> S <sub>0.01</sub> ) <sub>Σ=1.09</sub>                                       | Hessite |

Empirical formulae were calculated on the basis of total atoms (3 for hessite and 6 for petzite).

Most of the compositions of hessite from the Coranda-Hondol open pit are close to stoichiometric composition, with one exception. This grain shows a slight depletion in Ag and enrichment in Te (p6, Table 1, Fig. 1g). The silver and tellurium contents

of hessite range from 57.35 to 60.61 wt.%, respectively from 35.77 to 38.35 wt.%. In the Au-Ag-Te diagram this compositional variation (sample point 6, Table 1) was plotted between the theoretical composition of hessite and stützite (see Fig. 2b). In the case of petzite, the compositional variation shows a slight depletion in Te. The tellurium contents of petzite range from 32.31 (which is close to stoichiometric content) to 29.88 wt.%. Minor compositional variations of Ag and Au can be observed (Table 1).

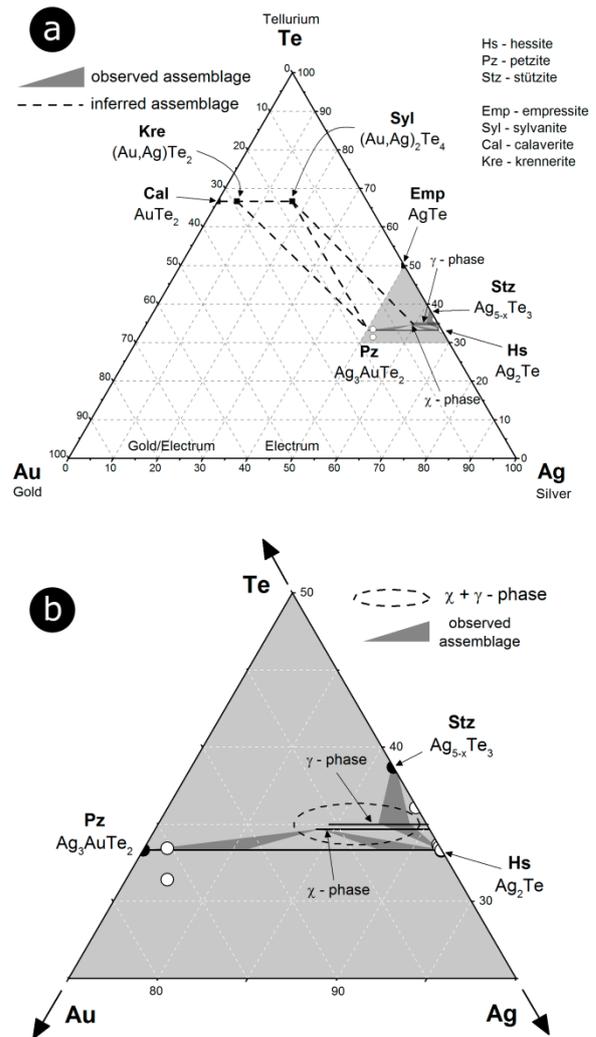


Figure 2. Ternary Au-Ag-Te diagram (atomic proportions) for mineral compositions analyzed in the present study (white circles) and for theoretical compositions of other associated Au-Ag tellurides (black circles)

The atomic percents of Au, Ag and Te are plotted in the ternary Au-Ag-Te diagram (Fig. 2). The plotted points filled with white color correspond to experimental data from this study, while the points filled with black color correspond to other Ag- and/or Au-Ag-tellurides of the system Au-Ag-Te.

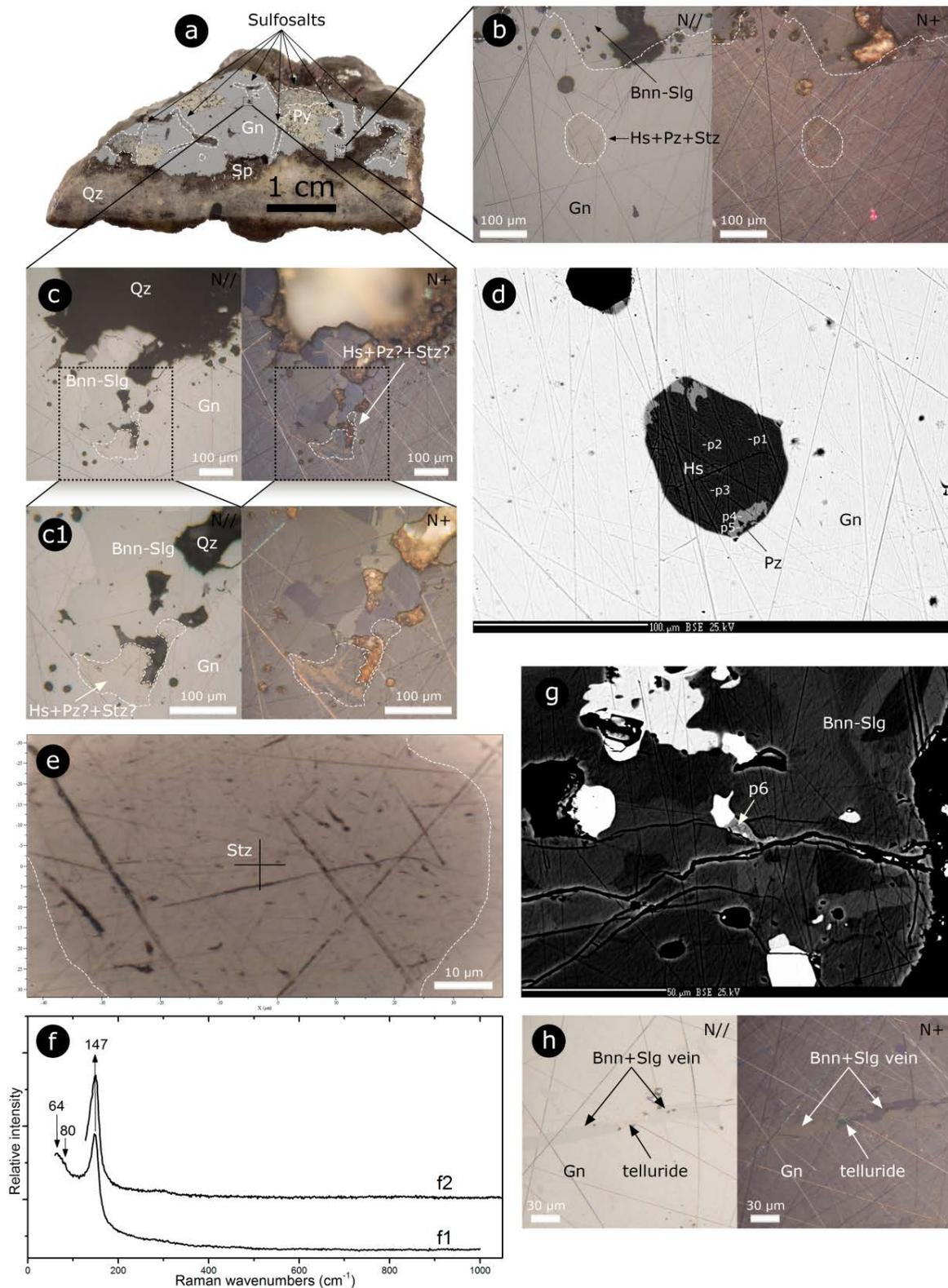


Figure 1. a) Polished section with quartz (Qz), galena (Gn), pyrite (Py), sphalerite (Sp) and veins of sulphosalts in galena; b) c) c1) Reflected light microphotographs (left – plane polarized light; right – cross polarised light) demonstrating the presence of telluride minerals found as inclusion in galena (Gn) and at the contact with bournonite-seligmanite (Bnn-Slg) and quartz (Qz); d) Back-scattered electron image (BEI) of petzite (Pz) found as inclusions in hessite (Hs); e) The crosshair marked in black is the spot where the micro-Raman spectrum of stützite (Stz) (Figure 1f, spectrum f1) was acquired and compared with reference R070701 (spectrum f2), (Downs, 2006); g) Back-scattered electron image (BEI) of hessite (Hs) found as inclusion in bournonite-seligmanite (Bnn-Slg); and h) Au-Ag-Te minerals in the veined sulfosalts (Bnn-Slg).

The solid lines represent the observed assemblage (hessite-petzite-stützite) in the Coranda-Hondol deposit, in terms of the system Au-Ag-Te. The dotted lines represent the inferred assemblages in the system Au-Ag-Te based on the association hessite-petzite-stützite and considering the ternary system (Cabri, 1965) and the binary system silver-tellurium (Kracek et al., 1966; Karakaya & Thompson, 1991).

### 4.3. $\mu$ -Raman spectroscopy

The Raman spectrum of petzite could not be acquired due to the small size of the grains of this mineral. Due to the fact that the sample was polished in order to remove the thin layer of carbon, the Raman spectrum acquired from the area does not correspond to hessite as it was first believed based on EPMA analyses, but it reveals the presence of stützite ( $\text{Ag}_{5-x}\text{Te}_3$ , where  $x = 0.24-0.36$ ). The stützite, hessite and petzite are intergrown irregularly, suggesting the contemporaneous formation (Burbank & Henderson, 1932).

The Raman spectrum of stützite is shown in the figure 1, spectrum f1, and it was compared with the reference standard (Fig. 1, spectrum f2, R070701, with the measured chemistry  $\text{Ag}_{4.94}\text{Te}_{3.00}$ , Downs (2006)). The Raman spectrum of stützite is in good agreement with the Raman spectrum of synthetic  $\text{Ag}_{5-x}\text{Te}_3$  (stützite) (Milenov et al., 2013).

According to the group theory classification (Aroyo et al., 2006a; Aroyo et al., 2006b), the full vibrational representations of  $\text{Ag}_{5-x}\text{Te}_3$  (stützite) contain  $\Gamma = 9A_{1g} + 3A_{1u} + 6A_{2g} + 10A_{2u} + 6B_{1g} + 8B_{1u} + 5B_{2g} + 8B_{2u} + 11E_{2u} + 15E_{2g} + 18E_{1u} + 11E_{1g}$  modes (space group  $P6/mmm$  (No. 191) and its point group  $D_{6h}$  (6/mmm), crystal structure from Downs and Hall-Wallace (2003)). The representation may be reduced to  $9A_{1g} + 15E_{2g} + 11E_{1g}$  Raman active modes.

The Raman peak centered at  $147\text{ cm}^{-1}$  (tentatively assigned to  $E_g$  vibration) with the full width at a half maximum (FWHM) around  $20\text{ cm}^{-1}$  is the distinguishable feature for the stützite against that of hessite. Unfortunately, the notch filter cuts the wavelengths below  $60\text{ cm}^{-1}$ . At these low wavelengths, a Raman peak can be observed at  $64\text{ cm}^{-1}$  with a shoulder located at  $80\text{ cm}^{-1}$  (tentatively assigned to  $A_g$  vibrations). There were no other Raman peaks in the higher frequency spectral domain (over  $200\text{ cm}^{-1}$ ) of the stützite. The broadness of the band centered at  $147\text{ cm}^{-1}$  indicates the non-homogeneous distribution.

In the case of the Raman spectrum of hessite two strong peaks arise at  $119\text{ cm}^{-1}$  and  $138\text{ cm}^{-1}$

(R060226, Downs (2006)). The Raman bands observed in the present study and those characteristic bands of stützite, hessite and petzite are summarized in Table 2.

### 4.4. Formation of hessite, petzite and stützite

Taking into account the strong heterogeneity (varying from dark orange to dark bluish grey in crossed nicols) under the microscope (see figure 1b, 1c and 1c1), the hessite is monoclinic rather than cubic (Xu et al., 2012). Also, it may be pointed out that hessite is formed below  $145\pm 3\text{ }^\circ\text{C}$ , because the transition of hessite from monoclinic to cubic (fcc, face-centered cubic) occurs at this temperature (Cabri, 1965; Kracek et al., 1966; Karakaya & Thompson, 1991).

Table 2. Raman bands, comparison between stützite, hessite, petzite and observed Raman peaks in this study.

| This study              | stützite                |                         | hessite                 |                         | petzite                 |
|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|
|                         | †                       | ‡                       | †                       | ‡                       | †                       |
| 64 <sup>vw</sup>        | n/a                     | n/a                     | n/a                     | n/a                     | n/a                     |
| 80 <sup>sh</sup>        | n/a                     | n/a                     | n/a                     | n/a                     | n/a                     |
| -                       | n/a                     | -                       | <b>119<sup>vs</sup></b> | <b>110<sup>vs</sup></b> | n/a                     |
| <b>147<sup>vs</sup></b> | <b>147<sup>vs</sup></b> | <b>151<sup>br</sup></b> | <b>138<sup>vs</sup></b> | <b>138<sup>vs</sup></b> | n/a                     |
| -                       | -                       | -                       | -                       | -                       | <b>163<sup>vs</sup></b> |
| -                       | -                       | -                       | -                       | -                       | 174 <sup>m</sup>        |
| -                       | -                       | -                       | -                       | -                       | $\sim 320^m$            |

Notes: † Downs (2006), ‡ Milenov et al., (2013) (synthetic); <sup>vs</sup> very strong; <sup>s</sup> strong; <sup>m</sup> medium; <sup>w</sup> weak; <sup>vw</sup> very weak; <sup>sh</sup> shoulder; <sup>br</sup> broad; n/a - not applicable (the wavelengths are missing).

Furthermore, the intergrowths between hessite-petzite-stützite may be a breakdown product of the  $\chi(\chi)$ -phase (below  $50\pm 20^\circ\text{C}$ ) and/or a decomposition of the  $\gamma(\gamma)$ -phase (below  $120\pm 15^\circ\text{C}$ ) (Kiukkola & Wagner, 1957; Cabri, 1965; Afifi et al., 1988; Ciobanu et al., 2004a). Echaeva & Osadchii (2009) confirm the  $\gamma$ -phase decomposition reactions by electromotive force (EMF) method, which form a mixture of hessite and stützite.

Similar assemblages involving Ag- and/or Au-Ag-tellurides are discussed previously, e.g. stützite-hessite-petzite and hessite-stützite in Colorado (Honea, 1964), hessite-petzite-stützite in Emperor (Pals & Spry, 2003), petzite-hessite-stützite in Săcărîmb (Ciobanu et al., 2004a) and hessite-petzite or hessite-petzite-sylvanite in Roşia Montană (Ciobanu et al., 2004b).

Obviously, these assemblages do not represent the initial conditions of ore-formation but rather a

re-equilibration during cooling. According to the previous discussions and taking into account the phase relations of Cabri (1965), the assemblage of hessite-petzite-stützite forms due to the breakdown of a mixture between  $\chi$ -phase and  $\gamma$ -phase at temperatures below  $120\pm 15$  °C.

The observations made regarding the Au-Ag- and/or Ag- tellurides assemblages from the Coranda-Hondol open pit suggest the contemporaneous formation. This can be separated into at least two substages: (i) an early gold-rich telluride-bearing substage and (ii) a later silver-rich telluride-bearing substage dominated by hessite and subordinated amounts of petzite and stützite. The presence of the early gold-rich telluride-bearing substage is still questionable and more analytical and optical observations need to be made to that effect. These assumptions imply the existence of another assemblage consisting of ?calaverite-?krennerite-?sylvanite, ?krennerite-petzite, ?sylvanite-hessite and/or ?sylvanite-petzite (see the inferred tie lines in figure 2a).

## 5. CONCLUSIONS

Ag- and/or Au-Ag-tellurides (hessite, petzite and stützite) are identified for the first time at the Coranda-Hondol open pit.

Optical and textural observations are accompanied by EPMA and  $\mu$ -Raman analysis for identification purpose. The  $\mu$ -Raman spectrum of stützite shows a distinct fingerprint against hessite, petzite or other Ag- and/or Au-Ag-telluride. The broadness of the main Raman peak of stützite ( $147\text{ cm}^{-1}$ ) may be related to the non-homogeneous distribution.

The Ag- and/or Au-Ag-telluride are represented by hessite with subordinated amounts of petzite and stützite. They usually occur as patches in galena, bournonite-seligmanite and at the contact between galena and bournonite-seligmanite series.

The microscopic observations and the presence of the intergrowths of hessite, petzite and stützite may be related to the breakdown of a mixture between  $\chi$ -phase and  $\gamma$ -phase of ternary Au-Ag-Te solution, below  $120\pm 15$  °C.

The observations of the hessite-petzite-stützite assemblage at the Coranda-Hondol open pit suggest that they have formed contemporaneously and that they can possibly be attributed to a later silver-rich telluride-bearing substage.

Further investigations are however necessary to interpret the presence of other assemblages (?calaverite-?krennerite-?sylvanite, ?krennerite-petzite, ?sylvanite-hessite and/or ?sylvanite-petzite)

which probably belong to a gold-rich telluride-bearing substage preceded by the later silver-rich telluride-bearing substage.

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