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"Invisible" gold and PGE elements in synthetic crystals of sphalerite and covellite: A EPMA, LA-ICP-MS and XAFS study

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Sphalerite and covellite are widespread minerals in the different genetic types of deposits and forms under the various conditions.
The purpose of this work is to determine the possible range of concentration and chemical state of Au and PGE (Pt, Pd, Rh) in sphalerite (Zn,Fe) S and covellite (CuS). These minerals were synthesized using gas transport and salt flux techniques. The crystals of ZnS were grown using the gas transport method at 850°C and the salt flux one using NaCl/KCl, CsCl/NaCl/KCl, and LiCl/RbCl eutectic mixtures at 850, 645 and 470°C, respectively. CuS crystals were synthesized using the salt flux method in RbCl/LiCl melt at 470 and 340°C. The trace metal activity was always controlled by the presence of pure metal or its sulfide, and, therefore, the concentration of these elements in synthesized phases represent the maximum possible value for given T/f(S2) synthesis parameters. The LA-ICP-MS and/or EPMA techniques were used to determine the Au concentration in synthesized phases. The concentration of Au in sphalerite, synthesized at 850°C with admixture of Cd, Se, In, Fe, and Mn, reached 0.3wt%, whereas the sphalerite cell parameter extremely increased up to 5.4161Å relatively to 5.4060 Å for pure ZnS. It was found that the observed high Au concentration is caused by the presence of In (2091±46 ppm Au in sample with Fe and In in comparison with 14±7 for Se-bearing ZnS, 94±12 ppm for Fe-Mn-bearing sphalerite, and 96±46 for Fe-bearing sphalerite. The concentration of Au in Fe-bearing sphalerite synthesized at 645°C does not exceed 5 ppm. Therefore, increase of temperature results in the increase of Au concentration in sphalerite.
The concentration of Au in another Fe-bearing-sphalerite series synthesized using gas transport method at 850°C various from 200 to 500 ppm and depends on the iron content. This fact could be related to the oxidation state or Fe in ZnS-FeS solid solution series. The concentration of Pt and Pd, Rh in sphalerite is below the detection limit of LA-ICP-MS (∼30 ppb). However, these trace elements change the cathodoluminescence properties of ZnS. The concentration or gold in covellite was determined by both LA-ICP-MS and EPMA techniques and the final values clearly fit together. The maximum concentration can be observed at 450° and equal to 0.3wt%. This value changes minor due to the increasing of the temperature. In principle, adding admixtures of In, Zn, Se, Cu, Sb, Bi did not affect on the concentration of Au. However, in experiment where sulfur is excessive and a mixture of In, Zn, Se, Cu, Sb, Bi, were added the concentration of Au is equal 0.128+0.028 ppm. The gold distribution in covellite and sphalerite is always homogeneous. According to XANES data, atoms of Au in the crystal structure covellite is in triangles, formed by the atoms of Cu. In sphalerite gold is in "invisible" state too.