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SEM/EDX AND AFM STUDY OF GOLD CEMENTATION ON COPPER(I) SULPHIDE

This paper is methodological in nature. The results presented here are a preliminary step towards finding an optimal method of sulphide surface preparation for systematic AFM studies of $AuCl_4^-$ cementation products. This technique makes it possible to see surface topography in nanoscale.

Products of $AuCl_4^-$ cementation on synthetic copper (I) sulphide, $Cu_{1.86}S$, were investigated by scanning electron microscopy (SEM/EDX) and atomic force microscopy (AFM). The microstructure of the sulphide surface before and after the reaction with $AuCl_4^-$ was analysed. Changes in the amount and shape of the products formed on polished plates and grains of the same material were compared. The process was performed in a flow-through vessel (on the plates) or in a circulation apparatus (on grain samples), by contacting the sulphide with aqueous solution containing $1.5 \cdot 10^{-4}$ mol/dm^3 $HAuCl_4$ and $1*10^{-1}$ mol/dm^3 HCl. The amount of gold deposited on a grain sample was determined from $AuCl_4^-$ concentration decay. The reaction progress on plates was evaluated from the intensities of the Au_L line of the EDX spectra. The maximum cementation degree was found to be equal to about 80 estimated atomic layers of metallic gold deposited on the sulphide. In SEM images the products were seen as crystallites of different sizes, between 0.1μm (the lowest limit detectable by SEM with a magnification of 5000x) and about 1.5μm, randomly distributed over heterogeneous surfaces of the plates and grains. The AFM technique made it possible to see the shapes of smaller crystallites, with their growth limited to the inside of cavities or cracks formed by polishing. The average density of the crystallites observed by AFM was of the order of 10 per 1μm^2 geometric surface area.

Key words: cuprous sulphide, copper (I) sulphide, sulphide surface preparation,
gold deposition/cementation, SEM/EDX and AFM techniques

INTRODUCTION

Polish copper-bearing ores contain economically significant gold admixtures (Piestrzyński i Wodzicki, 2000, Piestrzyński i Sawłowicz, 1999, Kucha et al., 1998). The gold contained in those ores accompanies much larger admixtures of silver, and is much more difficult to recover than the latter (Bortel et al., 1985). Gold recovery losses can reach even 50 percent of the original content, especially when the noble metal is chemically bound with the host mineral crystal lattice (Kucha et al., 1998).

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Considering the variety of forms of gold and silver occurrence in copper ores, optimal processing conditions must be selected for each ore lot. To optimize the recovery of metals occurring as accessory minerals, it is necessary to find forms in which they occur in copper-bearing ores, and the changes they undergo during processing (flotation, hydrometallurgical leaching).

In the literature there are no quantitative descriptions of the changes that the gold contained in copper sulphides undergoes. Much more information is available about reactions of silver with copper sulphides. The basic electrochemical property of noble metal ions is their spontaneous deposition (cementation) in the presence of less noble metals. Due to the significant differences in chemical properties of silver and gold (e.g., their reactivity with sulphur), it can be expected that cementation of these metals on copper sulphide will be significantly different, in both the preliminary and secondary stages of the process.

So far, gold cementation processes were mostly investigated on such sulphide minerals as piryte, pyrhotite, galena and sphalerite (Mycroft et al., 1995, Scaini et al., 1997, Becker and Hochella, 1996).

This paper refers to our earlier studies of noble metal (Ag, Au) cementation on non-stoichiometric copper (I) sulphide. Those earlier studies (Barzyk et al., 2002) were done on 60-75μm grains of $Cu_{1.86}S$, which made it possible to determine the rate of the process by measuring the decrease in noble metal concentration as a function of time (Ag^+ - by potentiometry, $AuCl_4^-$ - by UV-Vis spectrophotometry). The sulphide grain samples were also suitable for x-ray diffraction (XRD) studies, thus permitting crystalline phases to be determined in the cementation products (at concentrations greater than 2%, i.e., above the method's detectability). Another advantage of grain samples was that small grain quantities could be collected during the process, to be examined by the electron microscope (SEM/EDX) without altering the process conditions (Barzyk and Pomianowski, 2001). The grains proved, however, not suitable for AFM studies on a larger scale due to the high roughness of the surface. Sharp topographic outlines prevented the AFM tip's access to a significant portion of the surface and often damaged the tip, which significantly reduced the applicability of the AFM microscopy

This paper is methodological in nature. The results presented here are a preliminary step towards finding an optimal method of the copper(I) sulphide surface preparation for systematic AFM studies of topology of products of gold cementation. This technique makes it possible to see surface topography in nanoscale; consequently, structures with dimensions greater than, or comparable to the scanned area, cannot be studied by the AFM microscopy. It should be noted that in order to characterize the topography of the portion of surface that is measurable by AFM technique, a statistical approach is needed, which means that a statistically significant number of images (at least 21) must be analysed. In order to access what proportion of the actual surface is available to AFM microscopy, comparative studies should be done by techniques that can scan larger areas, e.g., electron microscopy (SEM).

It is possible to attain atomic resolution with the AFM technique in high vacuum if the examined surfaces are smooth in atomic scale, i.e., surfaces of monocrystals prepared in a way favouring the formation of atomic terraces (Dakkouri et al., 1999, Warren et. al., 2002). Preparation of monocrystals is costly and in the case of copper (I) sulphide – practically impossible. Also, a monocrystal is too ideal a model of mineral and thus it may lack properties that dominate the course of surface processes in real conditions. These processes are known to depend strongly on the number of defects and admixtures occurring in the mineral crystal lattice (Vaughan, et al., 1997).

Mineral samples are most often prepared in the form of polished plates - particularly suited for electrochemical techniques and solid-state methods (e.g., ESCA). It should be borne in mind that polishing with abrasives may lead to the formation of numerous surface defects – the effect may be much stronger than in the case of comminution. When materials are comminuted they usually break at points of the weakest bonds, i.e., along crystallographic planes with the strongest bonds (and the shortest interatomic distances). This favours the formation of states of lower energy than those in polished surfaces. On the other hand, polishing can lead to the formation of high-energy defects due to very high local stresses applied to microscopic areas. Defects created by polishing can increase the mineral's surface activity and affect its topography. On the other hand, topographic defects, typical of polished surfaces (e.g., scratches made by diamond paste with grain sizes below 3μm) will be outside the scanning capabilities of the AFM technique. Consequently, one should expect that topographic differences in the area "seen" by the AFM shall disappear, i.e., similar images should be obtained for plate and grains samples of the same mineral. The difference in the concentration of defects can, however, markedly affect the distribution of cementation products.

The objective of this study was to compare changes in the surface microstructure of polished plates and grains of the same copper sulphide ($Cu_{1.86}S$) produced by cementation of gold ions ($AuCl_4^-$) in diluted acid solution ($1x10^{-1}mol/dm^3$ HCl). The focus was on whether polishing markedly effects the number and distribution of defects as compared with the surface of grains (more representative of mineral processing).

EXPERIMENTAL

SURFACE ANALYSIS TECHNIQUES

A scanning electron microscope (XL 30) with a conventional secondary electron detector (for SEM) and an energy dispersive x-ray analyser (for EDX) were used to examine the surface microstructure and atomic composition of the surface region (in samples supported on a carbon tape). The electron beam penetrated the sub-surface region with a cross-section area of ca. 1 μm^2 and a depth of 2-4 μm.

The Atomic Force Microscopy (AFM) images were produced by a NanoScope E microscope (Digital Instruments) in the contact mode, with NanoProbes silicon nitride

cantilevers (force constant 0.12 N/m). An x-ray diffractometer (Dron 5) was used to obtain diffraction spectra (XRD) of the sulphide grains dispersed in nujol, using K_α the line of the copper lamp.

MATERIALS AND PROCEDURES

The samples of copper (I) sulphide ($Cu_{1.86}S$) were prepared in the form of grains or plates. The synthesis of $Cu_{1.86}S$, its comminution, classification, and the storage of the grains (class of 60-75 μm) was described earlier (Barzyk et al., 1981). The surface of 1g of the grains was $0.1m^2$, as determined by the BET method.

Plates with dimensions of 7x6x2 mm were used. Their surfaces were polished with abrasive paper (4000) and diamond paste with diamond grain size below 3 μm. After polishing, the plates were washed with CCl_4 and redistilled water. Cementation studies on plates were carried out in a flow-through vessel (Pomianowski and Barzyk, 1987). The solution was introduced to the vessel through a capillary (parallel to the plate) at a flow rate of about 60 ml/min. The solution containing $1.5 \cdot 10^{-4}$ mol/dm^3 $HAuCl_4$ in $1*10^{-1}$ mol/dm^3 HCl was in contact with the plate surface, and its concentration did not change throughout the experiment.

In order to study cementation of gold on grain samples, a circulation apparatus was designed, as described elsewhere (Barzyk et al., 2002). The initial concentration of the solution was $1.5*10^{-4}$ mol/dm^3 $HAuCl_4$ in $1*10^{-1}$ mol/dm^3 HCl (380 ml) and the content of gold ions gradually decayed as the reaction with sulphide grains (1g) progressed for less than 10 min. In order to deposit on the sulphide sample the amount of gold well detectable by x-ray diffraction (XRD), i.e., above 2 wt. percent per $Cu_{1.86}S$ grain sample, four portions of $HAuCl_4$ solution (with the above-specified composition) were added. Each subsequent portion was introduced after about 12 hours' equilibration with the oxygen-free electrolyte, circulating through the grain sample. (The step-by-step procedure is discussed in detail elsewhere; Barzyk et al., 2002). The amount of gold deposited on grain samples was determined from $AuCl_4^-$ concentration decay by UV Vis spectrophotometry (cf. Barzyk, 1990). The reaction progress on plates was assessed by comparing the intensities of the Au_L line of the EDX spectra obtained for the grains and plates.

Prior to the experiments, the surface oxidation products were removed from both the plate and grains by washing the mineral's surface with the stream of oxygen-free solution of $1 \cdot 10^{-1}$ mol/dm^3 H_2SO_4, according to the procedure described earlier (Pomianowski and Barzyk, 1987).

The solutions were prepared from analytical purity grade H_2SO_4, HCl, $HAuCl_4$, and double distilled water. In the experiments, industrial grade argon was used. The measurements were performed at room temperature, $20\pm3\,^oC$.

RESULTS AND DISCUSSION

The surface topography and composition of the sulphide, before and after the reaction with $AuCl_4^-$, are compared in Figs. 1-4, for four $Cu_{1.86}S$ samples differing in the extent of the cementation reaction. The results are presented in the form of sets of typical SEM and AFM images, EDX spectra and the corresponding AFM depth profiles. Figs. 1 a-d show the reference sample, i.e., the plate after the same pre-treatment as other plates, but not contacted with $AuCl_4^-$. The reference sample gives AFM images similar to those obtained earlier for grains of pure $Cu_{1.86}S$ (cf. Figs. 6b,c in Barzyk et al., 2002). Hence, the AFM pictures shown in Figs. 1c-d served as reference images for analysing the changes in the sulphide surface topography produced by cementation on both the plate and grain samples.
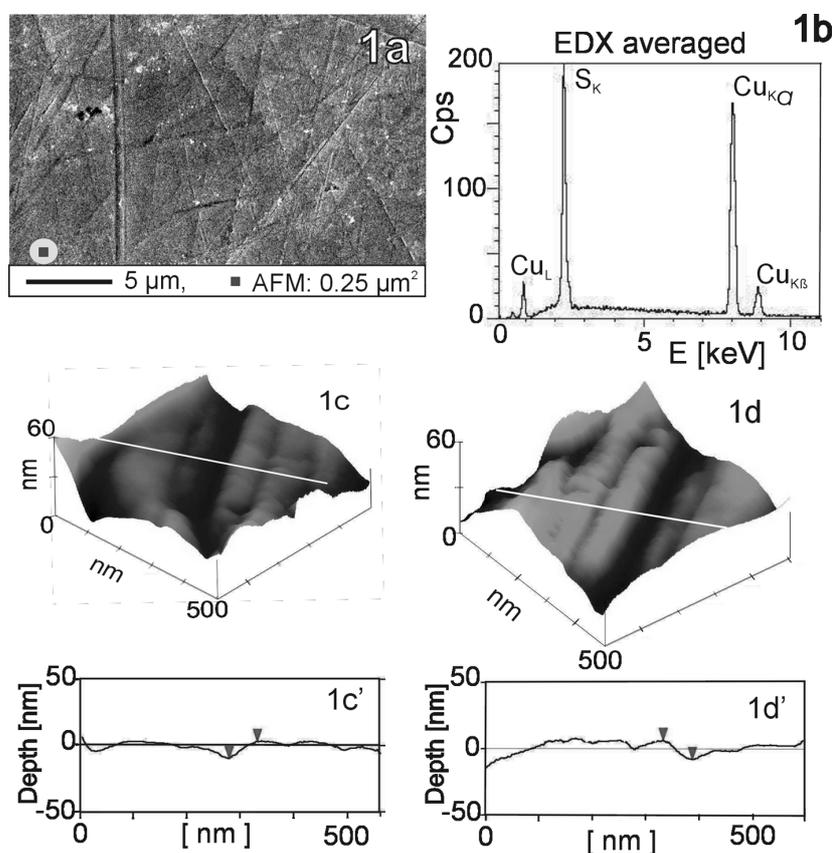
Figs. 1a-1d'. Surface microstructure of a $Cu_{1.86}S$ polished plate, after washing with $1 \cdot 10^{-1}$ mol/dm^3 H_2SO_4 oxygen-free solution for 30 min. (the preliminary stage); Fig. 1a – SEM image, Fig. 1b – averaged EDX spectrum registered along the area shown in the SEM image; Figs. 1c, 1d – AFM images typical of the sample, Figs. 1c-1d' – the AFM depth profiles
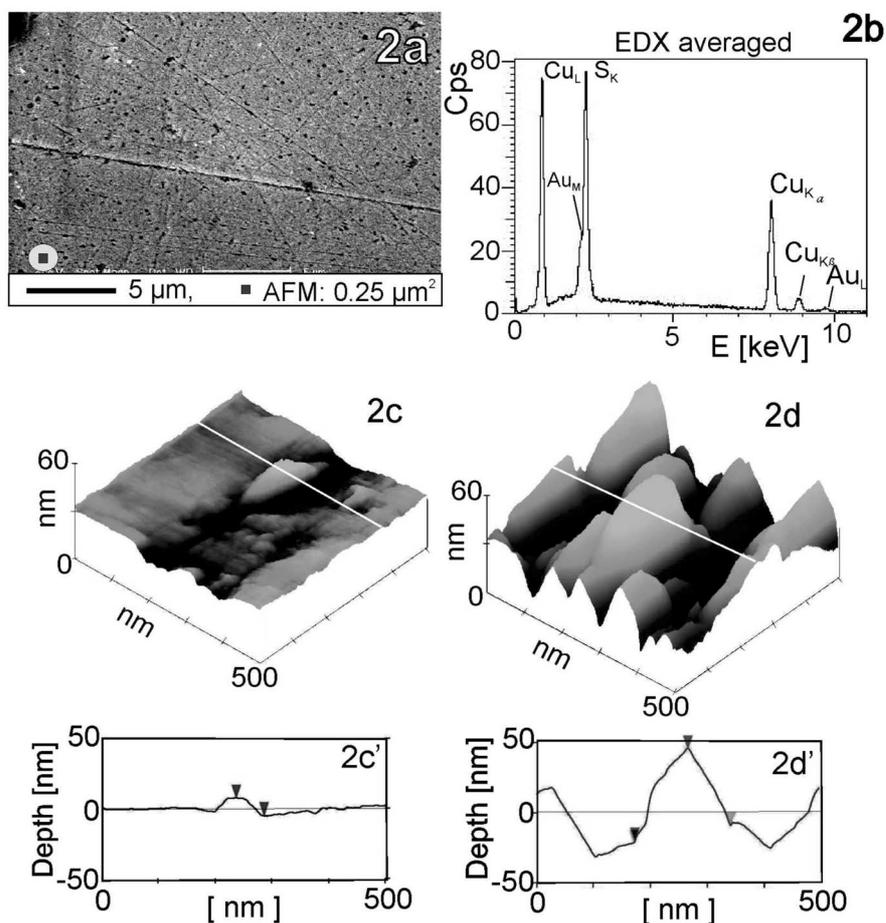
Figs. 2a-2d'. Surface microstructure of Cu$_{1.86}$S plate after 5 minutes reaction with air-saturated solution of 1.5· 10^{-4}mol/dm^3 HAuCl$_4$ in 1· 10^{-1} mol/dm^3 HCl (the part of the plate where the reaction progress was low due to partial screening of the surface by the capillary tip in the flow-through vessel); Fig. 2a – SEM image, Fig. 2b – averaged EDX spectrum registered along the area shown in the SEM image; Figs. 2c, 2d – typical AFM images; Figs. 2c', 2d' – the AFM depth profiles

Three of the samples discussed here in are in form of plates (Figs. 2-3). They are compared with the grain sample shown in Fig. 4. In the grain sample, the amount of gold deposited as a result of cementation was directly determined from the concentration decay. It corresponded to 3.4 wt. percent of Au per Cu$_{1.86}$S grain sample. The content of gold in the plates was estimated by comparing the corresponding averaged EDX spectra (i.e., the ratios of Au$_L$ to CuK$_\beta$ lines, whose energy values are shown in Table I) with the EDX spectrum of the grain sample. The comparisons show that the amounts of gold contained in the region penetrated by the

EDX technique are approximately 0.5 wt. percent (Fig. 2b) and 1 wt. percent (Fig. 3b). The content of gold was expressed in terms of the number of nominal atomic layers of the deposited metal, assuming that the average area occupied by a gold atom (at the BET surface) was 8.0Å^2, as found from the gold density ($19.3g/cm^3$). The calculated numbers of nominal layers in the samples treated with $AuCl_4^-$ (Figs. 2, 3, and 4) were about 15, 30 and 80, respectively. Note that the $Au_{M\alpha 1}$ line, much more intensive than the $Au_{L\alpha 1}$, could not be used for this quantitative estimation, as it is hidden in the $S_{K\alpha 1}$ line (cf. Table I and Figs. 2b, 3b, 4b). The only sample where the $Au_{M\alpha 1}$ line could be identified is presented in Fig. 4.
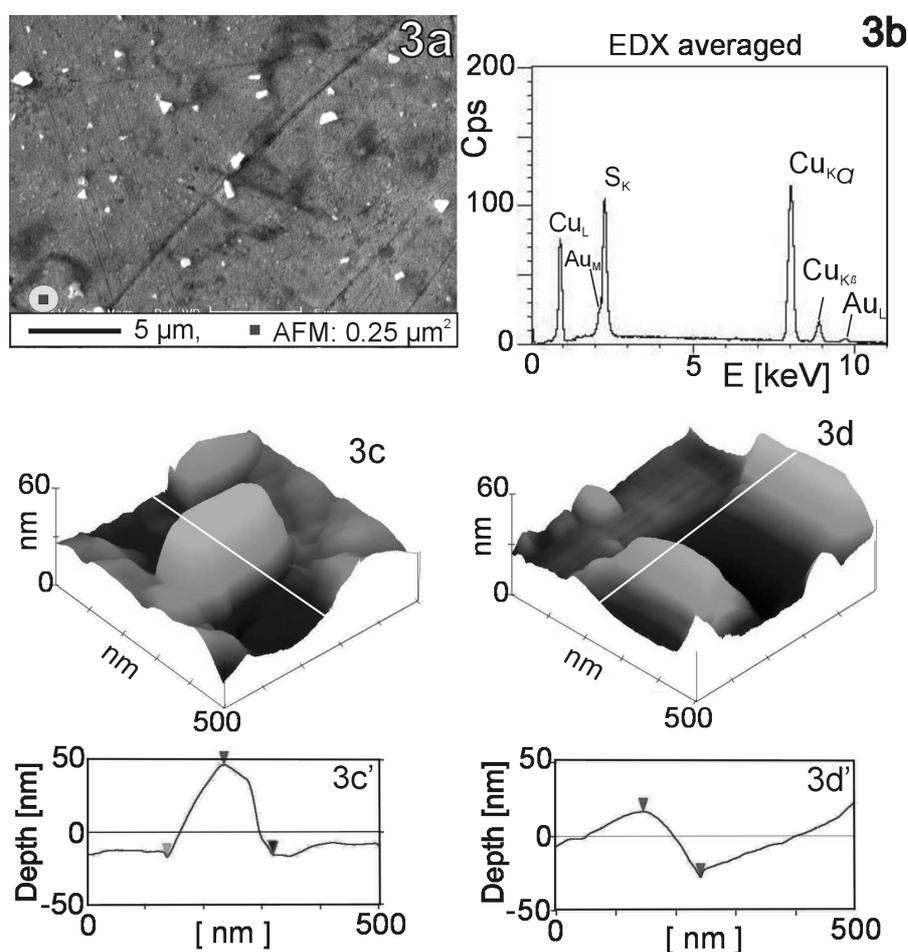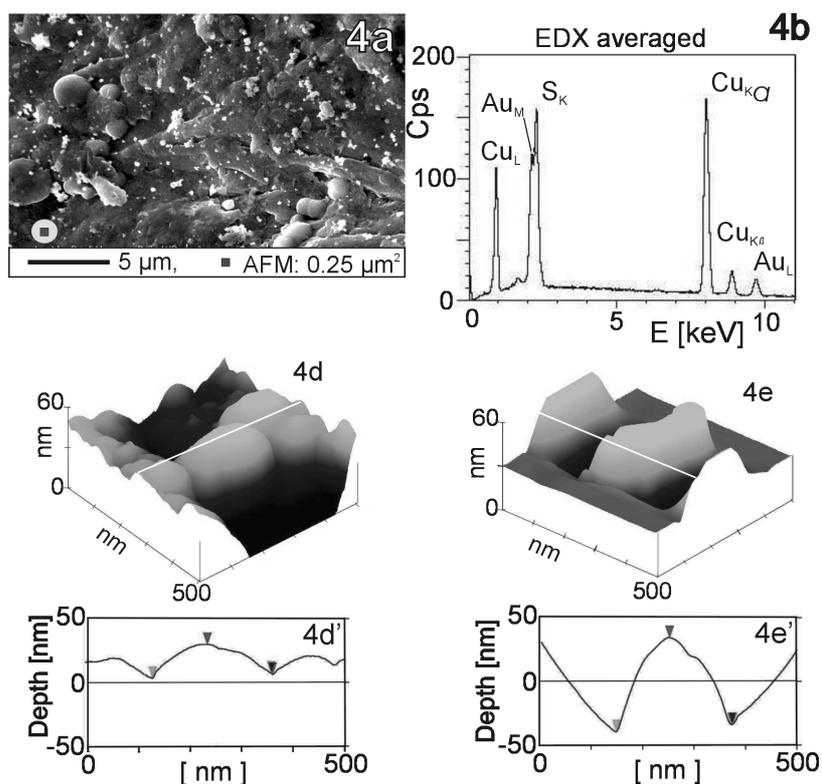

Fig. 3a-3d'. Surface microstructure of the area with a greater reaction progress, on the plate presented in Figs. 2a-d' (i.e., the part of the surface not screened by the capillary); Fig. 3a – typical SEM image, Fig. 3b - averaged EDX spectrum registered along the area shown in the SEM image, Figs. 3c, 3d – typical AFM images; Figs. 3c', 3d' – AFM depth profiles

Table I. The energy and associated wavelength of the strongest K, L, M lines of the elements
(Goodhew and Humphreys, 1988)

Element	Energy [keV]			
	$K\alpha_1$	$K_{\beta 1}$	$L\alpha_1$	$M\alpha_1$
S	2.31			
Cu	8.05	8.95	0.93	
Au	68.79		9.71	2.12

Figs. 4a-4e'. Surface microstructure of Cu $_{1.86}$S grains containing 4 wt. percent Au (i.e., after reacting with four portions of 1.5· 10^{-4} mol/dm^3 HAuCl$_4$ in 1· 10^{-1} mol/dm^3 HCl); Fig.4a – typical SEM image, Fig. 4b - averaged EDX spectrum registered along the area shown in the SEM image, Figs. 4d, 4e – typical AFM images; Figs. 4d', 4c' – AFM depth profiles

The cementation products can be seen in SEM images as crystallites of different size, i.e., from 0.1μm (which is close to the lower limit detectable in SEM of magnification of 5000x; cf. Fig. 2a) to about 1.5μm (cf. Figs. 3a, 4a). SEM did not reveal any preferential distribution of the product crystallites over the highly heterogeneous surfaces of plates and grains.

It is interesting that for pure sulphide (in the plate and grain samples) a characteristic layered texture is revealed by the AFM technique, i.e., layers with a thickness of about 100nm. The solid material seems to be preferentially split along the texture layers, which could be seen in numerous areas probed by AFM as shown in Figs. 1c-d (cf. also Figs. 6b-c of Barzyk et al., 2002). The layers were significantly disarranged by the cementation reaction, which could be seen as swelling and pitting of the layers, followed by their irregular growth - the effects shown in AFM images of plates and grains were characteristic of lower reaction progress (cf. Figs. 2c, d, and the results of Barzyk et al., 2002).

The degree of disarrangement of the original surface texture increased with the reaction progress, which is shown in the series of AFM images presented in Figs. 1c-d, 2c-d, 3c-d. At higher values of the reaction progress, isolated crystallites were observed in AFM images, on both the plates (Figs. 3c-d) and grains (Figs.4d-e). The smallest, crystallites revealed by the AFM technique are below $0.1\mu m$ (cf. Figs. 3c-d and 4d-e). They seem to be preferentially nucleated in textural cavities or cracks (probably formed by comminution) or in scratches formed on plates by polishing. The growth of the crystallites observed by the AFM technique looks to have been stopped by the opposite wall of the crack, or by other crystallite growing in the closest proximity (cf. Figs. 3c-c', 3d-d', 4d-d', 4e-e'). Generally 2-3 crystallites of dimension of 0.1-0.4 μm appeared in areas scanned by AFM (500 x 500nm), suggesting that the average density of nucleation centres is high (of an order of 10 per $1\mu m^2$ geometric surface area).

Crystalline products of the cementation process were identified by examining the grain samples by x-ray diffraction technique (XRD). The minimum content of gold detectable by XRD was of about 2 wt. percent Au per $Cu_{1.86}S$ grain sample, which is slightly lower than the gold content in the sample presented in Fig. 4. The crystalline products identified by the XRD method in the grain sample are mixture of Au^o, Au_2S and Au_2S_3 (cf. Barzyk et al., 2002). It cannot be ruled out that, at a lower gold content in the sulphide, the same products are formed in different ratio, which might be detected by applying a more sensitive method than XRD, e.g., the x-ray synchrotron techniques used by Warren et al., 2002. The occurrence of mixed products suggests a complex mechanism of the cementation process, involving secondary reactions between sulphur present on the surface and the gold deposited in the primary surface reaction. Furthermore, the surface processes may be accompanied by gold disproportionation reaction. (Mechanism of the cementation process will be discussed in detail, separately).

The cementation process appears to be accompanied by etching of the sulphide surface. These effects can be seen as pits (craters) with dimensions of about 0.3 μm on the plate containing about 0.5 wt. percent Au in the surface region (Fig. 2a). At the reaction progress corresponding to the content of about 1 wt. percent of Au (or to deposition of about 30 nominal layers of gold), the etching effects can be seen as cavities in the surface, noticeable in the SEM image in Fig. 3a. Pits that appear on

surface as a result of cementation can be attributed to the surface reaction releasing copper ions (I) from the sulphide as a co-product of the primary cementation reaction (initial cementation step). The cementation process involves the reduction of $AuCl_4^-$ ions to Au^o ad-atoms coupled with the oxidation of the sulphide copper to Cu^+ ions. Hence, cuprous ions should be released at the ratio of $AuCl_4^- : Cu^+ = 1:3$, however, the ratio is changed by the copper dis-proportionation reaction, $2Cu^+ \rightarrow Cu^0 + Cu^{2+}$, which is inherently accompanying processes at cuprous sulphide. The presence of Cl^- ions and oxygen dissolved in the solution may significantly increase the concentration of copper ions at the sulphide surface, resulting in enhanced etching effects.

CONCLUSIONS

1. The results of the studies employing mineral grains and flat surfaces are complementary. Grain samples permit cementation kinetics to be determined, whereas samples in the form of plates permit determination of the topology of the cementation products formed on the surface.
2. The size and distribution of crystallites formed on $Cu_{1.86}S$ as a result of the reaction with $AuCl_4^-$ were found to be similar for plates and grains.
3. The SEM images do not indicate any preferential distribution of Au cementation products on the copper (I) sulphide surfaces.
4. The AFM results confirm, however, that the smallest crystallites (0.1-0.4μm) are preferentially located within cracks, but this could be due to the limited space, which prevents their growth. The AFM results also suggest high density of the nucleation centres, about 10 per $1\mu m^2$, on both plates and grains.
5. Polished plates of copper sulphide may be used as convenient and representative samples for AFM study of gold cementation.

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Praca ma cel metodologiczny - optymalizację przygotowania próbek siarczku miedzi (I) do systematycznych badań topologii produktów cementacji jonów $AuCl_4^-$ na powierzchni, techniką AFM. Technika ta daje wgląd w topografię powierzchni w skali nanometrycznej. Badano produkty cementacji jonów $AuCl_4^-$ na syntetycznym siarczku miedzi (I) - $Cu_{1,86}S$, przy użyciu mikroskopii elektronowej (SEM/EDX) oraz mikroskopii sił atomowych (AFM). Porównano kształt oraz ilość produktów utworzonych na ziarnach i wypolerowanych płytkach z tego samego materiału. Proces cementacji prowadzono w naczyniu przepływowym (płytki) oraz w aparacie cyrkulacyjnym (próbki ziarn), przez kontaktowanie próbki w strumieniu roztworu zawierającego w 1 dm^3 wody $1,5 \cdot 10^{-4}$ mola $HAuCl_4$ oraz $1 \cdot 10^{-1}$ mola HCl. Ilość złota osadzonego na ziarnach siarczku w wyniku cementacji określono wyznaczając ilość jonów $AuCl_4^-$ ubywających z roztworu w czasie przebiegu procesu. Stopień zaawansowania przebiegu procesu na płytkach oszacowano przez porównanie intensywności linii Au_L widm EDX próbki ziarnowej i płytek. Badana maksymalna ilość produktów cementacji odpowiadała osadzeniu około 80-tu szacunkowych warstw atomowych Au na powierzchni. Produkty cementacji obserwowane na obrazach SEM mają kształt krystalitów o różnej wielkości, od około 0.1μm (dolna granica rozdzielczości SEM przy powiększeniu 5000 razy) do 1.5μm. Obrazy SEM nie wykazały preferencyjnego rozmieszczenia tych krystalitów na niejednorodnej powierzchni płytek lub ziaren.

Technika AFM umożliwiła obserwację kształtu małych krystalitów (0.1μm -0.4 μm), wskazując na ich preferencyjne usytuowanie w rysach lub szczelinach teksturalnych ziaren siarczku. Średnia gęstość krystalitów obserwowanych techniką AFM siarczku jest rzędu 10 na 1 μm^2 powierzchni geometrycznej siarczku.