

# SHORT COMMUNICATIONS

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## New data for nagyagite\*

### Nagyagite history

NAGYAGITE has remained poorly characterized since it was first described more than two centuries ago (as Nagiakerez) by Werner (1789). The name was subsequently modified to nagyagite by Haidinger (1845). The mineral was analysed by many of the chemists of the day, with varying degrees of success; the best analyses in the last century being those of Sipőcz (1885) and Hankó (1890), which, although possibly representing mixtures to some degree, nevertheless give an excellent approximation to the more recent analyses of Giusca (1937), and to modern day microprobe analyses of Stumpfl (1970) and Stanley (in Criddle and Stanley, 1986, 1993). Likewise, the crystallography of nagyagite has been the subject of much debate as to whether its symmetry is tetragonal (Gossner, 1935), monoclinic pseudotetragonal (Schrauf 1878), ortho-rhombic pseudotetragonal, or even triclinic (Schrauf, 1878).

The data presented here, when taken with the data given in Criddle and Stanley (1986, 1993), substantially characterize this mineral, although a full structural study remains to be accomplished. The soft platy nature of the mineral and the likely presence of twinning make it unlikely that material suitable for such a structural study will be easily found.

The specimen used in this study (BM 32941) is from the type locality of Nagyag, Transylvania (now Sacarimb, Romania).

### Single-crystal study

Two thin crystal fragments of nagyagite, megascopically free of any imperfections, were mounted and studied by precession single-crystal methods employing Zr-filtered Mo radiation. One crystal was mounted with  $101^*$  parallel to the dial axis, then remounted such that  $a^*$  was parallel to the dial axis. The other crystal was mounted with  $b^*$  parallel to the dial axis. The levels collected were:  $hk0 \rightarrow hk2$ ,  $h0l \rightarrow h2l$ ,  $0kl \rightarrow 2kl$  and  $101^* \wedge b^*$ . Precession photos are relatively good but neither crystal is suitable for a crystal structure determination.

Nagyagite is pseudo-tetragonal with orthorhombic symmetry. It is platy  $\{010\}$  with perfect  $\{010\}$  and excellent  $\{101\}$  cleavages. The unit-cell parameters, measured from precession zero-level films, are  $a$  8.336,  $b$  30.10,  $c$  8.290 Å. Systematic absences of the order,  $hkl$  with  $h + l \neq 2n$ ,  $hk0$  with  $h \neq 2n$ ,  $h0l$  with  $h + l \neq 2n$  and  $0kl$  with  $k \neq 2n$  and  $l \neq 2n$ , dictate that the permissible space-group choices are  $Bbmm(63)$ ,  $Bb2_1m(36)$  or  $Bbm2(40)$  (diffraction aspect  $Bb^{**}$ ). There is a pseudo-extinction of the type  $h0l$  with  $h \neq 2n$  and  $l \neq 2n$  that suggests pseudo-space groups  $Bbcm$  or  $Bba2$ . Reflections of the type  $hkl$  with  $h = l = 2n$

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TABLE 1. X-ray powder data for nagyagite (BM 32941)

$I_{\text{est}}$	$d\text{\AA}_{\text{meas.}}$	$d\text{\AA}_{\text{calc.}}$	$hkl$	$I_{\text{est}}$	$d\text{\AA}_{\text{meas.}}$	$d\text{\AA}_{\text{calc.}}$	$hkl$
20	15.0	15.10	020	10	2.321	2.321	282
5	5.06	5.08	131	10	2.208	2.213	292
5	3.78	5.03	060			2.091	400
		3.78	080	50	2.081	2.086	410
10	3.63	3.66	240			2.072	004
5	3.201	3.63	042	5	1.964	1.975	450
		3.216	260	5	1.914	1.913	0142
		3.199	062	10b	1.842	1.835	432
100	3.023	3.020	0100	30	1.823	1.823	2132
20	2.938	2.943	$2\bar{0}\bar{2}$	5	1.774	1.774	490
		2.930	212	30	1.710	1.709	0104
5	2.889	2.889	222	5	1.665	1.663	$4\bar{1}\bar{1}0$
80	2.829	2.825	232	5	1.519	1.521	$2\bar{1}\bar{7}2$
5	2.741	2.742	242	40	1.511	1.510	$0\bar{2}00$
20	2.646	2.646	252			1.472	404
5	2.536	2.541	262	30	1.473	1.470	414
40	2.431	2.431	272				

1. 114.6mm Debye-Scherrer powder camera; Cu radiation Ni filter (Cu-K $\alpha$  wavelength = 1.54178 Å)

2. b = broad line

3. Intensities estimated visually

4. Not corrected for shrinkage and no internal standard

5. Indexed on  $a = 8.363$ ,  $b = 30.20$ ,  $c = 8.288$  Å.

are very strong and predominate both on the single-crystal films and on the X-ray powder pattern. For such a large cell there is a dearth of resolvable powder reflections, and many of the stronger reflections have several possible indices. Under these conditions reliable unit-cell refinement is difficult. The refined unit-cell parameters  $a$  8.363(7),  $b$  30.20(1),  $c$  8.288(7) Å,  $V$  2093(2) Å<sup>3</sup>,  $a:b:c = 0.2769:1:0.2744$ , are based on measurements of 15 powder reflections representing  $d$ -values between 3.023 and 1.511Å for which unambiguous indexing was possible, based on visual inspection of precession single-crystal films. All permissible reflections down to 1.45Å were observed on precession films. A fully indexed powder pattern is given in Table 1.

#### Electron-microprobe analyses

The specimen which provided material for the single-crystal study was found to be chemically homogeneous (see Table 2); differences between the analyses 1 and 3 can be ascribed to different standardizations rather than true chemical variation. The results are comparable with most of the reported analyses. Through trial and error techniques, the best fit formula was one based

on 40 atoms. This yielded an ideal formula of  $\text{Pb}_{13}\text{Au}_2\text{Sb}_3\text{Te}_6\text{S}_{16}$ , which compares remarkably well to that given by Sipőcz (1885) of  $\text{Pb}_{28}\text{Au}_4\text{Te}_{14}\text{Sb}_6\text{S}_{34}$ . This latter formula has fallen out of general use in favour of the more recent  $\text{Pb}_5\text{Au}(\text{Te},\text{Sb})_4\text{S}_{5-8}$  (Palache *et al.*, 1944), or the formula given in Nickel and Nichols (1991) of  $\text{Au}(\text{Pb},\text{Sb},\text{Fe})_8(\text{Te},\text{S})_{11}$ ; presumably on the basis of Stumpfl's (1970) assertion that it would be more appropriate to group Te and S together.

Taking the density of nagyagite as given by Sipőcz (1885) (7.46), Hankó (1890) (7.35) and Frondel (in Palache *et al.*, 1944) (7.40), a calculation was made for the requirements of formula units (Z) for the cell given. For the ideal formula of  $\text{Pb}_{13}\text{Au}_2\text{Sb}_3\text{Te}_6\text{S}_{16}$  the calculated density with  $Z = 2$ , is 7.51 and for the empirical formulae given in Table 2, respectively 7.47 and 7.43 for columns 4 and 5. These calculated densities are in excellent agreement with the measured densities.

This is unlikely to be the end of the nagyagite story. Recent work by Spiridonov (1991), Udubasa *et al.* (1993) and Lupulescu *et al.* (1993) suggests a wider compositional variation in some analysed samples. Working out whether this variation can be accommodated by nagyagite *sensu stricto* or whether, in fact, there are possibly

TABLE 2. Electron-microprobe data for nagyagite

wt. %	1	2	3	4	5
S	10.83	10.52–11.04	11.07	15.95	16.11
Fe	0.02	0.00–0.08	0.01	0.01	0.01
Cu	0.02	0.00–0.12	0.04	0.01	0.03
Ag	0.10	0.00–0.35	0.03	0.04	0.01
Sb	7.68	7.55–8.19	7.76	2.98	2.97
Te	16.97	16.55–17.30	17.10	6.28	6.25
Au	7.47	6.87–7.99	7.66	1.79	1.82
Pb	56.76	55.86–57.53	56.83	12.94	12.80
Total	99.85		100.50	40	40

1. Average of 61 analyses on specimen BM 32941 used for the single-crystal study.
2. Ranges of the 61 analyses.
3. Average of 7 analyses on different area of specimen BM 32941.
4. No of atoms (sum to 40 atoms) of analysis 1.
5. No of atoms (sum to 40 atoms) of analysis 3.

Instrument: Cameca SX50

Operating conditions: 20 kV and 20 nA on Faraday cage

Standards: Pure metals, FeS, PbS.

several new minerals in a homologous series, as suggested by Francis *et al.* (1992), will require further study. Discussion on charge balance and the degree of metallic bonding in the mineral, also raised by Francis *et al.* (1992), must also await the availability of suitable crystals, particularly those amenable for crystal-structure study.

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## Post-recrystallisation phenomena in metamorphosed stratabound sulphide ores: a comment

RECENTLY, Vokes and Craig (1993) described and illustrated evidence for sequential mobilisation of sphalerite, chalcopyrite and quartz in the small Gressli deposit in central Norway during Caledonian amphibolite-facies metamorphism. These authors stressed the need to study other similarly deformed and metamorphosed deposits to determine if similar textures might be present. The classic Cu–(Zn) pyritic deposits at Sulitjelma in northern Norway (Cook *et al.*, 1990, 1993) are already well known as being excellent examples of stratiform massive sulphide deposits in which the effects of polyphase amphibolite grade metamorphism and deformation have contributed significantly to the observed textures within the sulphide assemblages. Extensive modification of the sulphide bodies during metamorphism and evidence for remobilisation within the large Giken II deposit in the centre of the orefield (Fig. 1) has been described by Cook *et al.* (1993). As well as

mobilisation of the more ductile sulphides (pyrrhotite, chalcopyrite), these processes have also played a significant role in the redistribution of precious metals within the deposit (Cook, 1992).

Continuing study of massive sulphide samples from this locality has revealed textures very similar to those described by Vokes and Craig (1993). The samples examined were collected from levels –371 and –396 near the eastern edge of the Giken II deposit, shortly after it closed in the summer of 1991. This area of the mine was known for copper contents higher than in other parts of the deposit and also for elevated concentrations of Ag, Au, Sb, As, Pb and Bi (Cook *et al.*, 1992). Abundant quartz veins cross-cut the stratabound sulphides and carry significant chalcopyrite, pyrrhotite and galena. This area was the source of samples in which widespread (re)mobilisation phenomena have been previously observed. Study of other