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Preparation and crystal structures of novel LaOAgS-type copper and silver fluoride chalcogenides

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Systematic studies of LaOAgS-type alkaline earth–Cu/Ag fluoride chalcogenides revealed six new compounds SrFCuTe, SrFAgS, SrFAgSe, SrFAgTe, BaFAgS, and BaFAgSe. Crystal structures have been determined for these as well as for BaFCuS. Based on trends in the bond distances within the PbO- and FeS-type layers, a semi-qualitative approach is suggested to predict new members of the LaOAgS family.

1. Introduction

The quaternary equiatomic LaOAgS structure type, recently termed the “1 1 1 1” structure, encounters now several hundreds of representatives [1], and their number is increasing rapidly. A variety of its representatives exhibit exciting properties such as ionic conductivity [2], transparency coupled with semiconductivity [3], and medium-temperature superconductivity [4]. Their two latter properties, as well as crystal chemical peculiarities have been reviewed recently [1,5,6]. The atomic arrangement is particularly simple and consists of two alternating *anti*-isotypic (PbO- and FeS-type) layers (Fig. 1). It is in fact its own *anti*-type [6] and hence a convenient object for crystal chemical analyses. Not all possible combinations of PbO- and FeS-type layers observed in related structures can however be coupled to yield this structure. For instance, all rare earths form $[Ln_2O_2]^{2+}$ fluorite slabs in the $[Ln_2O_2]Br_2$ oxide bromides [7] but no LaOAgS-type compound has hitherto been prepared for a Ln later than Er [8,9]. Similarly, some *anti*-fluorite slabs, *e.g.* $[Mn_2Bi_2]^{2-}$, have been observed in related *anti*-PbFCl (or CeFeSi, “1 1 1”) [10,11] or $HfCuSi_2$-type [12] structures but as yet not in any “1 1 1 1”-like compound. A semi-quantitative approach to description of “crystal chemical boundaries” of the structure type has been suggested in [13,14]. Both layers are comprised of tetragonally distorted (local symmetry $\bar{4}2m$) tetrahedra (Fig. 1) so that all center-to-vertex distances remain equal but six edges and bond angles split into 4 + 2 groups. For both tetrahedra, the a_2 edges equal each other and the a cell parameter, and the structure is supposed to be stable until a limiting deformation is reached which may, for instance, be expressed in terms of differences between edge lengths or bond angles of ideal and distorted tetrahedra. Such considerations find some “eigenvalues” common for several isostructural rare-earth series but they suggest the bond length to be invariable when proceeding from one structure to another. This is true, for instance, for metal–chalcogen bonds within isostructural series of rare-earth–coinage metal oxide chalcogenides where $[T_2Ch_2]^{2-}$ layers (T = Cu or Ag, Ch = chalcogen) reside in very similar chemical environment, but such transferability of bond lengths had not been systematically addressed among other chemical families of LaOAgS-type compounds. A second relatively large family of such compounds containing the same $[T_2Ch_2]^{2-}$ layers is fluorides of divalent metals, $A^{II}FTCh$, where A = Sr, Ba, Sm, and Eu [15–19]. Most of these compounds have been characterized structurally, yet some combinations of A, T, and Ch remain unaddressed. In the current paper, we attempt to compare the structural data for LaOTCh, SrFTCh, and BaFTCh compounds. To obtain a more complete data set, the yet missing compounds SrFCuTe, SrFAgCh (Ch = S, Se, Te), BaFAgS, and BaFAgSe were addressed. As no structural data had been presented for the known compounds BaFCuS [17] and LaOAgTe [14], they have also been included into this study.

2. Experimental

2.1. Synthesis

Most compounds in question were prepared starting from mixtures of powdered AeCh, AeF_2, and T_2Ch (or Ae, AeF_2, T, and Ch). All operations were performed in an argon-filled M’Braun dry box (H_2O/O_2 less than 1 ppm). In a typical run, the mixtures were pressed into pellets and annealed in evacuated ($2–5 \times 10^{-1}$ Torr) carbon-coated silica capsules at 600–650 °C with several re-grindings. When alkaline-earth

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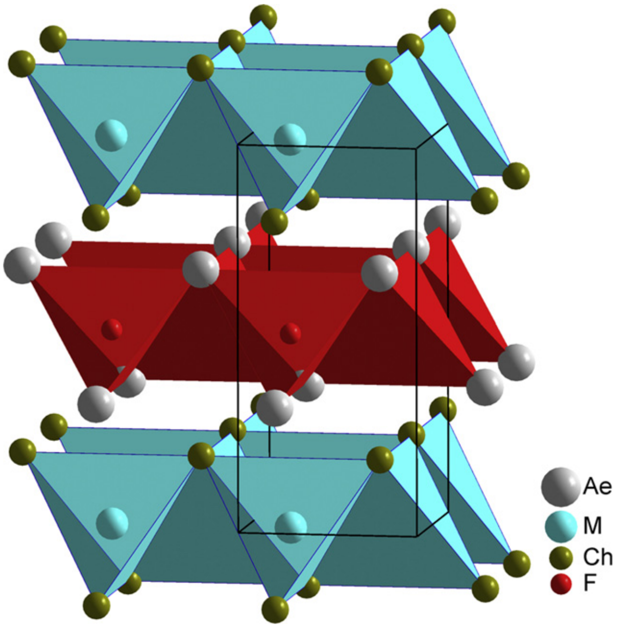

Fig. 1. The structure of LaOAgS-type AeFTCh fluoride chalcogenides (Ae = Sr or Ba; T = Cu or Ag, and Ch = S, Se, or Te).

metals were used as precursors, the heating rate of the first annealing was very low and several plateaus were used to control the exothermic reaction between the metal and the chalcogen. The compounds SrFAgS and BaFAgS were prepared in a way similar to preparation of LaOAgS [2]: 1:1:1 mixtures of AeS, AeF_2 (A = Sr or Ba), and Ag_2S were ground (not pressed) and heated at 600 °C for 48–120 h in the presence of iodine vapor as a catalyst. Exact synthesis conditions are collected in Table 1. Full details of all refinements are provided in the Supplementary material.

XRD data indicated formation of the known compound BaFCuS as well as six new compounds, SrFCuTe, SrFAgS, SrFAgSe, SrFAgTe, BaFAgS, and BaFAgSe, all belonging to the LaOAgS ("1111") type. All new compounds were prepared nearly free from by-phases except SrFAgS where the maximal yield was about 40%. Longer annealing of SrFAgS-containing samples with iodine vapors led to decomposition of the target compound with liberation of metallic silver. Attempts to prepare LaOAgTe under various conditions below 700 °C led to poorly crystallized phase-impure samples, so the structure refinement was not feasible.

2.2. Structure determination

Powder X-ray diffraction data were collected on a Bruker D8 Advance diffractometer (Cu $K\alpha_1$ radiation, Ge(1 1 1) monochromator, reflection geometry) Rietveld refinements were performed with the TOPAS package [20] using the fundamental parameter approach as reflection profiles. Preferred orientation was corrected using a spherical harmonic approach developed in TOPAS. Further experimental details, refined atomic parameters, and main structural parameters are collected in Tables 2–4. For SrFAgS, the multi-phase refinement was less accurate and we consider the data more cautiously. A typical Rietveld refinement plots (for the case of BaFAgS) is shown in Fig. 2.

3. Results and discussion

Our results demonstrate that all AeFTCh compounds with Ae = Sr, Ba; T = Cu, Ag, and Ch = S, Se, and Te, can be prepared using standard solid-state synthesis, except AeFAgS where, akin to LaOAgS, presence of iodine catalyst is necessary. Compared to LnOTCh analogs, synthesis of AeFTCh requires milder conditions (shorter annealing times and lower temperatures) but a long preliminary step.

Comparison of the Ln–O and Ae–F families reveals that while the LnOTCh series (obtained by fixing the composition of the *anti*-fluorite layer and varying the rare-earth metal) terminate at various rare-earths (CuS:Eu [8]; CuSe:Er [8]; CuTe:Nd [8]; AgS:Ce [20], AgSe:Nd [14]; AgTe:La [14]), all possible AeFTCh compositions exist for Ae = Sr and Ba. As EuFCuCh exhibit almost the same cell parameters and bond distances as SrFTCh (Table 3), analogous EuFAgCh compounds could possibly exist though it is difficult to estimate their stability towards redox decomposition into Eu^{III}FCh and Ag^0. However, in a yet another rare-earth representative of the fluoride family, SmFCuSe [19], Sm^{2+} and Cu^+ surprisingly appear redox-compatible within the same compound, so the question if more Ln^{II} fluoride chalcogenides can exist is open for further studies.

The structural data for LaOTCh, SrFTCh, and BaFTCh are summarized in Table 4 (except LaOAgTe). In addition, structural information for EuFCuCh is also included. Even a cursory survey shows that all bond lengths (La—O, T—Ch, or Ae—F) are not constant within the family though the variations are mostly below 2–3% which seems to be the accuracy limit of semi-empirical geometrical approaches addressed above [13,14]. Both La—O and Ae—F bonds

Table 1
Synthesis conditions for the new fluoride chalcogenides.

Compound	Precursors	Annealing procedure	Phase content[a], %
SrFCuTe	Sr, SrF_2, Cu, Te	Slow heating to 600 °C, grinding, pelletizing and firing twice at 600 °C for 48 h	SrFCuTe – 86, SrF_2 – 5, CuTe – 9
SrFAgS	SrS, SrF_2, Ag_2S	Annealing the powder mixture at 600 °C twice for 120 h in the presence of I_2 vapors	SrFAgS – 43, SrF_2 – 22, Ag – 23, SrS – 12
SrFAgSe	Sr, SrF_2, Ag, Se	Slow heating to 600 °C, grinding, pelletizing and firing twice at 600 °C for 48 h	SrFAgSe – 96, SrF_2 – 4
SrFAgTe	Sr, SrF_2, Ag, Te	Slow heating to 600 °C, grinding, pelletizing and firing twice at 600 °C for 48 h	SrFAgTe – 85, SrF_2 – 9, $SrCO_3$ – 6
BaFCuS	BaS, BaF_2, Cu, S	Slow heating to 600 °C, grinding, pelletizing and firing twice at 600 °C for 48 hs	BaFCuS – 98, BaF_2 – 2
BaFAgS	BaS, BaF_2, Ag_2S	Annealing the powder mixture at 600 °C for 48 h in the presence of I_2 vapors	BaFAgS – 94, BaF_2 – 3, $BaSO_4$ – 3
BaFAgSe	BaSe, BaF_2, Ag, Se	Slow heating to 600 °C, grinding, pelletizing and firing twice at 600 °C for 48 h	BaFAgSe – 94, BaF_2 – 2, $BaSeO_4$ – 2

[a] According to Rietveld refinements.

Table 2
Details of structural experiments.

Compound	SrFCuTe	SrFAgS	SrFAgSe	SrFAgTe	BaFCuS	BaFAgS	BaFAgSe
Crystal system	Tetragonal						
Space group	*P*4/*nmm* (# 129)						
Z	2						
a, Å	4.2474(1)	4.0593(1)	4.1652(1)	4.3397(1)	4.1229(1)	4.2406(1)	4.3449(1)
c, Å	9.2003(2)	9.1521(2)	9.2552(2)	9.5947(2)	9.0063(2)	9.3029(2)	9.4018(1)
V, $Å^3$	165.98(1)	150.81(1)	160.57(1)	180.70(1)	153.09(1)	167.29(1)	177.49(1)
2θ range	18–95	18–100	18–90	15–95	18–80	8–110	8–110
R_p	0.062	0.109	0.052	0.070	0.034	0.027	0.023
R_{wp}	0.082	0.142	0.070	0.097	0.045	0.035	0.031
R_B	0.015	0.047	0.011	0.028	0.022	0.007	0.007
R_e	0.065	0.082	0.056	0.060	0.033	0.031	0.019

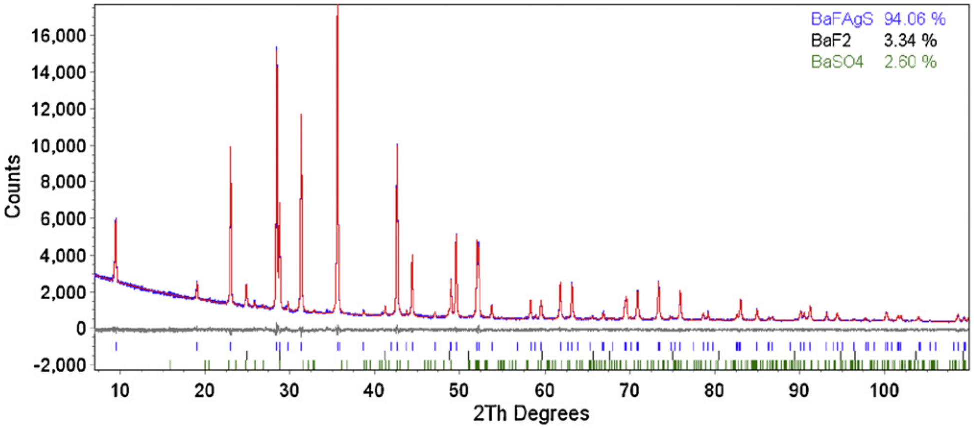

Fig. 2. Final Rietveld refinement plot for BaFAgS.

elongate when passing from sulfides to selenides and tellurides (suggesting T = const) or from copper to silver compounds (suggesting Ch = const), the increments being non-sensitive to the nature of the coinage metal or chalcogen, respectively, within the same 2–3%. Based on these increments, we estimated the bond lengths in LaOAgTe as well as in the yet unknown compounds EuFAgCh (these data are given in italics in Table 4).

The variations of T–Ch bonds among LaOTCh, SrFTCh (T = Cu, Ag), and EuFTCh (T = Cu) compounds are marginal since $d(\text{La–O}) \approx d(\text{Sr–F}) \approx d(\text{Eu–F})$. Larger changes are observed when passing to BaFTCh as the Ba–F bond is sufficiently longer. Thus, the constant T–X bond lengths (T = transition metal, X = chalcogen or pnicogen) within the known LnOTX series are likely due to very smooth and limited variation of the environment of the $[T_2X_2]^{2-}$ layers when passing along the rare-earth series.

It noteworthy that while $d(\text{La–O}) \approx d(\text{Sr–F})$, SrFAgTe can be easily prepared nearly single-phase but not LaOAgTe. These compounds correspond to the largest mismatches in size of $AgTe_4$ and FSr_4/OLa_4 tetrahedra forming the FeSe and PbO-type layers, respectively, and the adjusting deformations of these layers required for a commensurate structure are also maximal. However, the predominantly ionic $[Sr_2F_2]^{2+}$ layers are much “softer” to bond angle deformations compared to more covalent $[La_2O_2]^{2+}$ units; thus the structure of LaOAgTe appears to be much more strained compared to SrFAgTe. The edge lengths of regular $AgTe_4$ tetrahedra (calculated from $d(\text{Ag–Te}) = 2.879$ Å, see Table 4) should equal $a^\circ = 4.70$ Å which should be compared with the corresponding cell parameters, $a(\text{SrFAgTe}) = 4.34$ Å and $a(\text{LaOAgTe}) = 4.23$Å [14]. The deformation of the $AgTe_4$ tetrahedra (expressed in terms of $a^\circ - a$ [13]) is 0.36 Å in SrFAgTe *vs.* 0.47 Å in LaOAgTe. For BaFAgTe, the deformation of $AgTe_4$ tetrahedra drops to 0.17 Å. A more general comparison of the *a* cell parameters of the AFTCh compounds (A = Eu, Sr, Ba) with the corresponding minimal values (termination points) for the LnOTCh series (CuS: 3.874 Å, CuSe: 3.865 Å [8]; CuTe: 4.040 Å [22]; AgS: 3.926 Å [21]; AgSe: 4.028 Å, AgTe: 4.233 Å [14]) clearly indicates that deformations of the $[T_2Ch_2]^{2-}$ layers in the AFTCh are much smaller, so existence and relatively easy preparation of the fluoride chalcogenides is not surprising. The only exception yet not clearly understood is SrFAgS where the reason probably lies in low reactivity of both SrS and Ag_2S. Note that $CeOAg_{1-x}S$ had also been prepared with difficulty and a rather low yield [21], and a ternary compound $BaAg_2S_2$ can be obtained only via an indirect way [23].

Considering 0.40 ± 0.05 Å as the maximal possible deformation of the $[T_2Ch_2]^{2-}$ layers, we estimated existence of some more AeFTCh compounds. The largest value of *a* cell parameters for structures involving $[A_2F_2]^{2+}$ layers is observed for AFI fluoride iodides (A = Eu and Sr: 4.25 Å; A = Ba: 4.69 Å [24,25]). Adding 0.40 Å to these values yields the maximal edge lengths of regular TCh_4 tetrahedra of ~4.6 Å (Eu and Sr) and ~5.0 Å (Ba) and bond lengths of ~2.8 Å and ~3 Å, respectively. This suggests existence of more “exotic” chalcogenides in this family. Comparison of bond lengths in sphalerite-type MTe and MPo compounds (M = Be, Zn, Cd [26–29]) reveals $d(\text{M–Po}) \approx d(\text{M–Te}) + 0.1$ Å. This yields the estimate bond lengths of $d(\text{Cu–Po}) \approx 2.8$ Å and $d(\text{Ag–Po}) \approx 3$ Å which coincide very well with the limiting values calculated above. Hence, SrFCuPo, EuFCuPo, BaFCuPo, and BaFAgPo can possibly be prepared, more likely than LaOCuPo. All other compositions are significantly less likely. Indeed, further increase of the FA_4 tetrahedra enhances

Table 3
Atomic coordinates for the novel fluoride chalcogenides.

	SrFCuTe		SrFAgS		SrFAgSe		SrFAgTe	
	z	*B*	*z*	*B*	*z*	*B*	*z*	*B*
Ae (¼ ¼ *z*)	0.1454(1)	1.05(7)	0.1534(2)	1.0[a]	0.1500(1)	0.5(2)	0.1358(2)	0.46(9)
F (¼ ¾ 0)		1.0[a]		1.0[a]		0.7(2)		1.0[a]
Ch (¼ ¼ *z*)	0.6758(1)	1.48(7)	0.6945(7)	1.0[a]	0.6959(1)	0.3(2)	0.6976(1)	0.52(8)
T (¼ ¾ ½)		2.08(8)		1.0[a]		1.4(2)		1.52(9)

	BaFCuS		BaFAgS		BaFAgSe	
	z	*B*	*z*	*B*	*z*	*B*
Ae (¼ ¼ *z*)	0.1772(1)	1.5(2)	0.1665(1)	1.16(1)	0.1598(1)	0.80(2)
F (¼ ¾ 0)		1.0[a]		1.2(1)		0.5(1)
Ch (¼ ¼ *z*)	0.6498(6)	1.4(2)	0.6760(3)	1.36(6)	0.6798(1)	0.83(3)
T (¼ ¾ ½)		2.7(2)		1.97(2)		1.75(2)

[a] Not refined.

Table 4
Bond distances (Å) and angles (°) for LaOAgS-type coinage metal oxide/fluoride chalcogenides.

Compound	LaOCuS[a]	LaOCuSe[a]	LaOCuTe[a]	LaOAgS[b]	LaOAgSe[c]	LaOAgTe
d(La—O)	2.360(1)	2.376(1)	2.402(1)	2.378(1)	2.388(1)	*2.415*
d(La—Ch)	3.256(1)	3.329(1)	3.487(1)	3.261(2)	3.337(1)	*3.500*
d(T—Ch)	2.429(1)	2.524(1)	2.656(1)	2.690(3)	2.754(1)	*2.880*
α_2(LaOLa)	115.58(1)	117.71(1)	120.95(2)	117.52(4)	119.44(2)	
α_4(LaOLa)	106.51(1)	105.51(1)	104.06(2)	105.60(3)	104.73(2)	
α_2(ChTCh)	110.59(5)	107.38(2)	103.80(2)	98.22(7)	96.97(2)	
α_4(ChTCh)	108.92(5)	110.53(2)	112.38(2)	115.37(7)	116.06(2)	
Compound	SrFCuS[d]	SrFCuSe[d]	SrFCuTe	SrFAgS	SrFAgSe	SrFAgTe
d(Sr—F)	2.445(1)	2.476(1)	2.515(1)	2.468(2)	2.503(1)	2.543(1)
d(Sr—Ch)	3.144(2)	3.239(2)	3.427(1)	3.190(3)	3.272(1)	3.456(1)
d(T—Ch)	2.430(2)	2.531(2)	2.671(1)	2.699(5)	2.761(1)	2.871(1)
α_2(SrFSr)	108.00(2)	110.26(4)	115.32(7)	108.88(5)	112.64(5)	117.1(1)
α_4(SrFSr)	110.21(2)	109.08(4)	106.63(3)	110.67(5)	107.91(2)	105.79(5)
α_2(ChTCh)	108.95(5)	106.75(5)	105.39(5)	97.50(1)	97.92(4)	98.19(5)
α_4(ChTCh)	109.73(5)	110.85(5)	111.55(3)	115.77(1)	115.54(2)	115.39(3)
Compound	BaFCuS	BaFCuSe[e]	BaFCuTe[f]	BaFAgS	BaFAgSe	BaFAgTe[g]
d(Ba—F)	2.607(1)	2.620(1)	2.652(1)	2.626(1)	2.642(1)	2.668(1)
d(Ba—Ch)	3.305(2)	3.387(1)	3.561(1)	3.337(2)	3.422(1)	3.586(1)
d(T—Ch)	2.464(3)	2.565(1)	2.694(1)	2.679(2)	2.753(1)	2.879(1)
α_2(BaFBa)	104.50(4)	108.01(3)	113.25(3)	107.69(2)	110.65(3)	116.15(1)
α_4(BaFBa)	112.01(2)	110.21(3)	107.61(3)	110.37(1)	108.88(1)	106.24(1)
α_2(ChTCh)	107.4(1)	111.44(3)	110.62(3)	104.65(8)	104.22(5)	103.69(1)
α_4(ChTCh)	113.6(2)	108.50(3)	108.90(3)	111.93(4)	112.16(2)	112.43(1)
Compound	EuFCuS[d]	EuFCuSe[d]	EuFCuTe[g]	EuFAgS	EuFAgSe	EuFAgTe
d(Eu—F)	2.465(2)	2.488(2)	2.520(1)	*2.488*	*2.515*	*2.587*
d(Eu—Ch)	3.134(2)	3.223(2)	3.393(1)	*3.180*	*3.256*	*3.422*
d(T—Ch)	2.434(1)	2.521(1)	2.666(1)	*2.700*	*2.780*	*2.869*
α_2(EuFEu)	106.44(5)	109.21(5)	114.40(1)			
α_4(EuFEu)	111.01(5)	109.60(5)	107.06(1)			
α_2(ChTCh)	108.46(2)	107.15(3)	105.27(1)			
α_4(ChTCh)	109.98(2)	110.64(3)	111.61(1)			

[a] Ref. [21].
[b] Ref. [2].
[c] Ref. [14].
[d] Ref. [16].
[e] Ref. [17].
[f] Ref. [18].
[g] Ref. [19].

the probability significantly, so we predict all RaFTCh (including Ch = Po) compounds to exist. Among oxide compounds, existence of all AcOTCh compounds is also very likely.

It is evident that the geometrical approach should be further tested by applying to other LaOAgS-type families, in particular, other chalcogenides of divalent rare-earths as well as isolobal related zinc, cadmium, and manganese pnictides. Several such compounds have been reported [30–32] but systematic investigations have yet not been done and are planned for the nearest future.

4. Conclusions

Our investigations have added several new representatives to the family of LaOAgS-type alkaline earth–coinage metal compounds, and existence of several new representatives involving the heaviest chalcogen and alkaline earth is considered to be probable based on geometrical considerations. Crystal chemical analysis of two LaOAgS-type families, LaOTCh and AeFTCh (Ae = Sr and Ba) indicates some interesting features. The deformations adjusting the layers into a commensurate structure and softness/hardness of these layers to such deformations play a key role in the structure stability. Using relatively soft $[Ae_2F_2]^{2+}$ fluorite layers together with $[T_2Ch_2]^{2-}$ counterparts results in larger number of representatives, as well as significant softening of synthesis conditions, as compared to related series involving more rigid $[Ln_2O_2]^{2+}$ moieties. The situation should become even more restricted if one would turn to $[M_2N_2]^{2+}$ layers (observed for M = Th [33], U [34], and Ce [35]). The accuracy of geometrical approaches to structure stability based on bond lengths is estimated to be about 2–3%. These considerations may appear of importance when new compounds of given composition and expected properties are aimed.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jallcom.2011.11.151.

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