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Subsolidus Phase Relations of the SrO-In₂O₃-CuO System in Air

J.-C. Grivel and K. Thyden

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The subsolidus phase relations of the SrO-In₂O₃-CuO system were investigated at 900 °C in air. Under these conditions, five binary oxide phases are stable: Sr₂CuO₃, SrCuO₂, Sr₁₄Cu₂₄O₄₁, In₂Cu₂O₅ and SrIn₂O₄. The pseudo-ternary section is characterised by six three-phase regions and is dominated by the SrIn₂O₄ phase, which is in equilibrium with all other phases. No Sr equivalents of the Ca₈In₂Cu₄O₁₅, Ca₉In₁₄Cu₂O₃₂, Ba₂In₂CuO₆, Ba₂InCuO_{4.5} or Ba₃In₄Cu₃O₁₂ phases were formed.

Keywords CuO, In₂O₃, phase equilibria, SrO, x-ray diffraction

1. Introduction

After the discovery of superconducting compounds with high critical temperature (T_c) in the Tl-Ba-Ca-Cu-O system,^[1] attempts have been made to replace thallium by harmless elements, in particular indium, which is located just above Tl in the periodic table. However, as reported by Hur et al.^[2] the solubility limit of In in the phase having the highest T_c is limited to about 20 at.% and In actually results in partial suppression of the superconducting properties. In contrast, superconductivity was observed in the In-based cuprates In_{0.3}Pb_{0.7}Ca_{0.8}Y_{0.2}Sr₂Cu₂O_y and (Bi,In)Sr₂(Gd,Ca)Cu₂O_y with T_c values of 60 and 42 K respectively.^[3,4] The effect of In substitution in a variety of superconducting cuprates differs significantly depending on the particular compound. In Bi₂Sr₂Ca₂Cu₃O₁₀, HgBa₂CaCu₂O₆ and (La,Gd)CaBa₃-Cu₇O_z, In doping is detrimental to the superconducting properties.^[5-7] In contrast, in YBa₂Cu₃O₇, a small amount of In substitution for Y appears to increase T_c slightly,^[8] whereas it can even induce superconductivity in (Nd,Ce)₂CuO_{4-y}.^[9] Interestingly, superconductivity can be induced by Zn doping in In₂O₃.^[10] The synthesis of a layered cuprate with composition reminiscent to that of some superconducting cuprates, i.e. In₂Ba₂CuO_{6-δ}, was also reported^[11] but this compound appears to be an insulator.

Whereas the phase equilibria of the pseudo-ternary systems CaO-In₂O₃-CuO and BaO-In₂O₃-CuO have been published,^[12,13] we are not aware of reports on the SrO-In₂O₃-CuO system. We have therefore determined the phase equilibria in the latter system in view of the possible occurrence of new ternary oxide phases as well as in order to provide a comparison with related systems.

1.1 Previous Work

Under ambient pressure conditions, In₂O₃ crystallises in the *Ia-3* space group (cubic).^[14] Several studies^[15-20] have been devoted to the SrO-CuO system, in which three phases are known to be stable at 800 °C in air: Sr₂CuO₃ (orthorhombic, space group *Immm*^[21]), SrCuO₂ (orthorhombic, space group *Cmcm*^[21]) and Sr₁₄Cu₂₄O₄₁, which is sometimes described as Sr₃Cu₅O₈ (orthorhombic, space group *Fmmm*^[22]). Under conditions of lower oxygen partial pressure, an additional binary oxide with SrCu₂O₂ composition was observed.^[20,23] The SrCu₂O₃ and Sr₂Cu₃O₅ phases were synthesised under high pressure (1.7-8 GPa).^[24]

According to the BaO-In₂O₃-CuO and CaO-In₂O₃-CuO phase equilibrium studies published by Horyń et al.^[12] and Godzhieva et al.^[13] respectively, in air in the temperature range 900-950 °C, the In₂O₃-CuO pseudo binary system contains only one binary oxide phase: In₂Cu₂O₅. The structure of this compound has been refined by Freund and Müller-Buschbaum^[25] as orthorhombic (space group *P2₁nb*). In₂CuO₄ single crystals with tetragonal symmetry were grown from a melt consisting of a 0.9BaO:0.1-KO_{0.5}:2SrO:4CuO nominal composition by cooling from 1150 to 300 °C.^[26] This phase does not appear in the above mentioned equilibrium diagrams.^[12,13]

Partial data on the SrO-In₂O₃ system can be found in the In₂O₃-TiO₂-SrO phase diagram section established by Li et al.^[27] at 1350 °C in air. Under these conditions, a single binary oxide compound is formed: SrIn₂O₄. The structure of this phase (orthorhombic, space group *Pnam*) was reported by von Schenk and Müller-Buschbaum.^[28] Sr₂In₂O₅ is only stable at higher temperatures.^[29]

In the SrO-In₂O₃-CuO system, no ternary oxide phase has been reported so far.

2. Experimental Details

High purity In₂O₃, SrCO₃ and CuO powders (all ≥99.9%) were thoroughly mixed in an agate mortar and calcined at 900 °C for 60 h. In total, 36 nominal compositions were

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Table 1 Phases observed in selected samples after equilibration and crystallographic data for the majority phases

Nominal cation ratio In:Sr:Cu	Phases (XRD) ^a	Space group ^b	Lattice parameters ^c			
			<i>a</i> , Å	<i>b</i> , Å	<i>c</i> , Å	β , °
0:0:100	CuO	<i>C2/c</i>	4.692(10)	3.425(2)	5.124(13)	99.52(3)
1:0:99	CuO (In ₂ Cu ₂ O ₅)	<i>C2/c</i>	4.694(11)	3.422(2)	5.124(15)	99.54(4)
49:0:51	In ₂ Cu ₂ O ₅ (CuO)	<i>P2₁nb</i>	12.291(5)	10.538(4)	3.279(2)	...
50:0:50	In ₂ Cu ₂ O ₅	<i>P2₁nb</i>	12.298(4)	10.547(3)	3.281(1)	...
51:0:49	In ₂ Cu ₂ O ₅ (In ₂ O ₃)	<i>P2₁nb</i>	12.296(3)	10.538(3)	3.279(1)	...
50:1:49	In ₂ Cu ₂ O ₅ (In ₂ O ₃ , SrIn ₂ O ₄)	<i>P2₁nb</i>	12.307(7)	10.549(5)	3.280(2)	...
49:1:50	In ₂ Cu ₂ O ₅ (CuO, SrIn ₂ O ₄)	<i>P2₁nb</i>	12.306(7)	10.552(5)	3.282(2)	...
99:1:0	In ₂ O ₃ (SrIn ₂ O ₄)	<i>Ia-3</i>	10.117(4)
100:0:0	In ₂ O ₃	<i>Ia-3</i>	10.118(3)
99:0:1	In ₂ O ₃ (In ₂ Cu ₂ O ₅) ^d	<i>Ia-3</i>	10.114(3)
98:0:2	In ₂ O ₃ (In ₂ Cu ₂ O ₅)	<i>Ia-3</i>	10.121(3)
68:32:0	SrIn ₂ O ₄ (In ₂ O ₃)	<i>Pnam</i>	9.842(5)	11.494(4)	3.261(3)	...
2/3:1/3:0	SrIn ₂ O ₄	<i>Pnam</i>	9.833(3)	11.491(2)	3.265(1)	...
66:34:0	SrIn ₂ O ₄ (SrO)	<i>Pnam</i>	9.839(4)	11.490(4)	3.264(2)	...
65.67:33.33:1	SrIn ₂ O ₄ (Sr ₁₄ Cu ₂₄ O ₄₁)	<i>Pnam</i>	9.834(4)	11.499(3)	3.264(2)	...
66.67:32.33:1	SrIn ₂ O ₄ (In ₂ O ₃ , In ₂ Cu ₂ O ₅)	<i>Pnam</i>	9.832(5)	11.494(5)	3.263(3)	...
1.00:66.67:32.33	Sr ₂ CuO ₃ (SrIn ₂ O ₄ , SrO)	<i>Immm</i>	12.701(1)	3.912(1)	3.499(1)	...
0.00:66.67:33.33	Sr ₂ CuO ₃	<i>Immm</i>	12.704(1)	3.913(1)	3.500(1)	...
1.00:65.67:33.33	Sr ₂ CuO ₃ (SrIn ₂ O ₄ , SrCuO ₂)	<i>Immm</i>	12.701(1)	3.912(1)	3.499(1)	...
1:50:49	SrCuO ₂ (SrIn ₂ O ₄ , Sr ₂ CuO ₃)	<i>Cmcm</i>	3.572(1)	16.328(3)	3.913(2)	...
0:50:50	SrCuO ₂	<i>Cmcm</i>	3.572(1)	16.325(2)	3.914(2)	...
1:49:50	SrCuO ₂ (SrIn ₂ O ₄ , Sr ₁₄ Cu ₂₄ O ₄₁)	<i>Cmcm</i>	3.571(1)	16.322(2)	3.912(2)	...
0.5:14.0:23.5	Sr ₁₄ Cu ₂₄ O ₄₁ (SrIn ₂ O ₄ , SrCuO ₂)	<i>Cccm</i>	11.464(3)	13.400(1)	27.64(1)	...
0.0:14.0:24.0	Sr ₁₄ Cu ₂₄ O ₄₁	<i>Cccm</i>	11.465(8)	13.401(3)	27.64(2)	...
0.5:13.5:24.0	Sr ₁₄ Cu ₂₄ O ₄₁ (CuO, SrIn ₂ O ₄)	<i>Cccm</i>	11.473(6)	13.405(3)	27.62(2)	...

^aPhases between brackets are minority phases

^bSpace group of the majority phase

^cThe lattice parameters are those of the majority phase

^dDetected by SEM/EDX but not by XRD

studied. After grinding, pellets were pressed under a pressure of 1.8 kbar and sintered at 900 °C for 60 h. Additional heat treatments under similar conditions with intermediate grinding and pressing were performed until equilibrium was reached. The samples were air-quenched at the end of the sintering treatments. All heat treatments were performed in air.

The phase content of the pellets was checked after each heat treatment by x-ray diffraction (XRD) in a STOE diffractometer using Cu-K_α radiation. Equilibrium was considered as achieved if no differences were detectable in the XRD patterns collected after two consecutive sintering treatments. Silicon powder was added as an internal standard for lattice parameter determinations in selected samples using the UnitCell leastsquares refinement programme.^[30] Scanning electron microscopy (SEM) and local compositional analysis were performed in a TM3000 Tabletop Microscope from HITACHI equipped with a QUANTAX 70 EDS analysis system.

3. Results and Discussion

3.1 SrO-CuO

Three binary oxide phases were formed at 900 °C in air: Sr₂CuO₃, SrCuO₂ and Sr₁₄Cu₂₄O₄₁, in agreement with previous reports.^[15-20] The SrCu₂O₂ phase was not found in the present study as a consequence of the lack of stability of this phase in air.^[20,23] The high-pressure phases SrCu₂O₃ and Sr₂Cu₃O₅^[24] were not formed either.

3.2 In₂O₃-CuO

In agreement with the results presented in Ref 12 and 13, In₂Cu₂O₅ is the only binary oxide phase stable at 900 °C in air in this system. The detection of In₂Cu₂O₅ in the sample with Cu_{0.99}In_{0.01} nominal cation composition, coupled with the similar lattice parameters for CuO in this sample and in

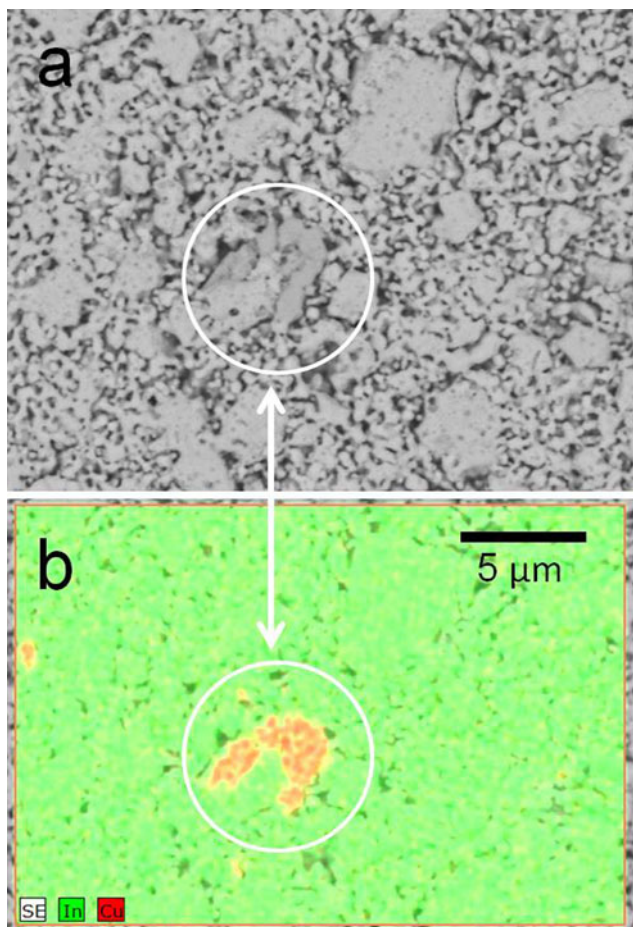


Fig. 1 SEM pictures [(a) backscattering mode and (b) EDS elemental map] showing evidence for coexistence of $\text{In}_2\text{Cu}_2\text{O}_5$ and In_2O_3 in the sample with $\text{In}_{0.99}\text{Cu}_{0.01}\text{O}_y$ nominal composition. The black areas are due to porosity. The encircled particle has an In:Cu atomic ratio of $53 \pm 3:47 \pm 3$

the pure CuO sample (Table 1) allow concluding that no extended substitution of In occurs in CuO under the present experimental conditions. The situation of the In_2O_3 rich part of the system is more intricate. In the works of Horyń et al.^[12] and Godzhieva et al.,^[13] no solid state solubility of Cu in In_2O_3 was reported. However, according to a recent report by Li et al.,^[31] 1 at.% Cu can be introduced in In_2O_3 . Using InCl_3 and CuCl_2 precursors and annealing at 900 °C in pure O_2 , Sasaki et al.^[32] even claim that single phase samples are obtained for a nominal composition described as $\text{In}_2\text{O}_3:\text{Cu}$. In the present study, $\text{In}_2\text{Cu}_2\text{O}_5$ impurities are detected by XRD only in samples with $x \geq 0.02$ in $\text{In}_{1-x}\text{Cu}_x\text{O}_y$ nominal compositions, whereas the sample with overall stoichiometry corresponding to a In:Cu = 99:1 atomic ratio appears to be single phase. However, the lattice parameter of the In_2O_3 phase is not affected within the accuracy limit of our measurements. Due to the limited resolution of x-ray diffraction, microscopical observations were performed on the “ $\text{In}_{0.99}\text{Cu}_{0.01}\text{O}_y$ ” sample. As shown in Fig. 1, particles characterised by a slightly darker contrast in backscattering mode are present in the sample. EDS

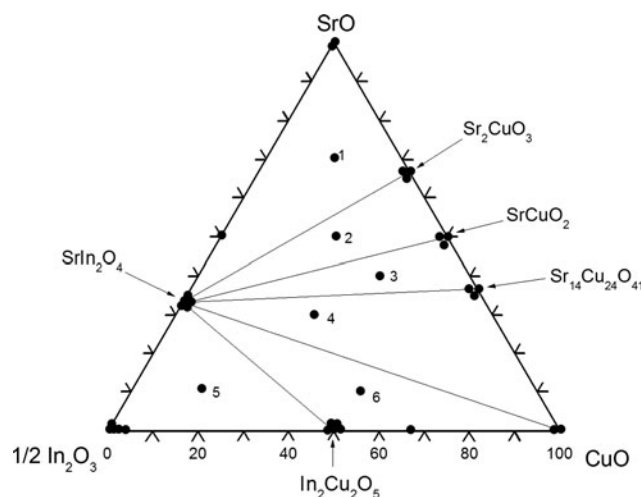


Fig. 2 Phase equilibria of the SrO- In_2O_3 -CuO system at 900 °C in air. Dots represent the studied compositions. The numbers 1-6 refer to the XRD patterns that are shown in Fig. 3

analysis on particles with sizes larger than 1 μm consistently yield an In:Cu atomic ratio close to that corresponding to $\text{In}_2\text{Cu}_2\text{O}_5$ (In:Cu = $53 \pm 3:47 \pm 3$). These particles are dispersed in an In_2O_3 matrix. We therefore conclude that Cu substitution in In_2O_3 must be limited to a value significantly lower than 1 at.% under the present experimental conditions, in agreement with the data published in Ref 12 and 13. As for the $\text{In}_2\text{Cu}_2\text{O}_5$ phase, it appears to be stoichiometric as deviations from the ideal composition result in equilibrium with either In_2O_3 (In excess) or CuO (Cu excess) as detailed in Table 1.

3.3 In_2O_3 -SrO

In this pseudo-binary system, SrIn_2O_4 is the only binary oxide phase stable under the present conditions.

Due to the strong tendency of SrO to form $\text{Sr}(\text{OH})_2 \cdot \text{H}_2\text{O}$ upon contact with air, we could not perform accurate lattice parameter determinations in the SrO-rich part of the diagram. However, the clear presence of SrIn_2O_4 as inferred by XRD analysis in the sample with $\text{Sr}_{0.99}\text{In}_{0.01}\text{O}_x$ nominal composition, suggests that the solubility of In in SrO is rather low as previously published,^[27] however at a significantly higher temperature (1350 °C). There is no evidence for Sr substitution in In_2O_3 , as SrIn_2O_4 is detected in a sample with $\text{In}_{0.99}\text{Sr}_{0.01}\text{O}_y$ nominal composition and the lattice parameters of In_2O_3 are not affected by the presence of Sr (Table 1). Likewise, the SrIn_2O_4 phase appears to be stoichiometric.

3.4 SrO- In_2O_3 -CuO

The subsolidus tie-line compatibilities of phases in the pseudo-ternary SrO- In_2O_3 -CuO phase diagram in air at 900 °C are shown in Fig. 2. There are five binary oxide phases: Sr_2CuO_3 , SrCuO_2 , $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$, $\text{In}_2\text{Cu}_2\text{O}_5$ and SrIn_2O_4 but no ternary oxide phases. All of the six three-phase regions are in contact with the SrIn_2O_4 phase, which can thus be in equilibrium with any of the other compounds

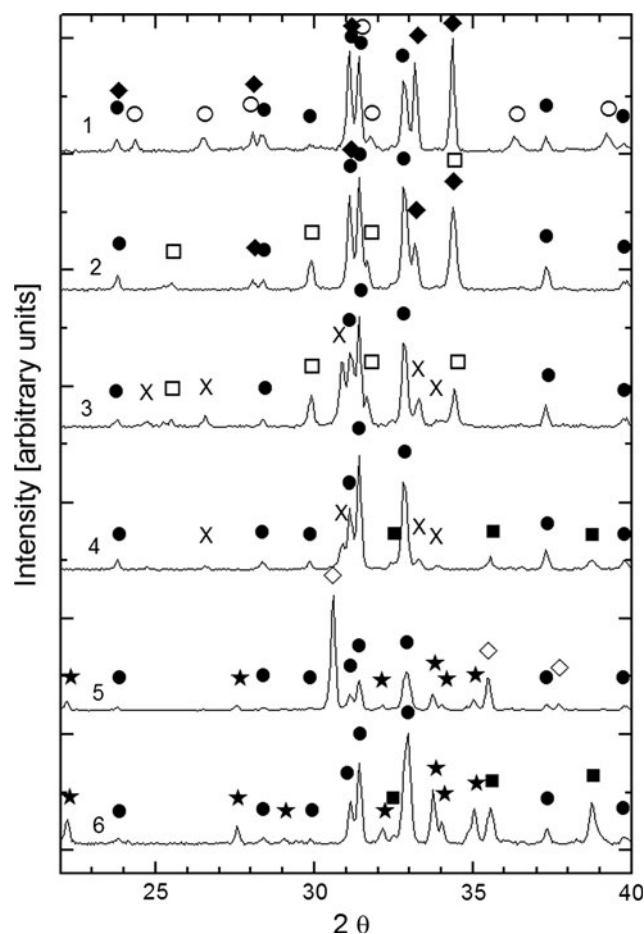


Fig. 3 Representative XRD patterns collected on samples with compositions lying in each of the three-phase regions of the pseudo-ternary sections. The pattern numbers refer to Fig. 2. Phases: ● SrIn_2O_4 , ○ $\text{Sr}(\text{OH})_2 \cdot \text{H}_2\text{O}$, ◆ Sr_2CuO_3 , □ SrCuO_2 , X $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$, ■ CuO , ◇ In_2O_3 and * $\text{In}_2\text{Cu}_2\text{O}_5$

depending on the overall composition. Representative XRD patterns for nominal compositions situated in all three-phase regions are shown in Fig. 3. None of the binary oxide phases of the pseudo-binary systems appear to give rise to ternary solid solutions as can be deduced from the appearance of 2-phase or 3-phase equilibria when attempts were made to dope these compounds with the element of the opposite corner (Table 1). The structure of this ternary section is similar to that of the $\text{SrO-RE}_2\text{O}_3\text{-CuO}$ systems where RE = Sc, Y, Lu, Yb, Tm and Er.^[33-37] A general trend of the $\text{SrO-Ln}_2\text{O}_3\text{-CuO}$ (Ln = lanthanide) is that the solubility of the lanthanide element in the $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$ phase decreases as the Ln^{3+} ion radius decreases.^[38] The radius of the In^{3+} ion is smaller than that of Lu^{3+} .^[39] It is therefore not surprising that no In solid solubility is observed in $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$.

4. Conclusion

In air, at 900 °C, the $\text{SrO-In}_2\text{O}_3\text{-CuO}$ system contains six three-phase regions and five binary oxide phases (Sr_2CuO_3 ,

SrCuO_2 , $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$, $\text{In}_2\text{Cu}_2\text{O}_5$ and SrIn_2O_4). No ternary oxide phases were formed under the present equilibration conditions. The structure of this ternary section is similar to that of the $\text{SrO-RE}_2\text{O}_3\text{-CuO}$ systems where RE = Sc, Y, Lu, Yb, Tm and Er, but without $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$ -based solid solution.

Acknowledgments

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