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Study and Characterization of $(K_xSr_{1-x})CrO_z$, x = 0.10-0.50 System

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Abstract: Studies of thermodynamic, electric and magnetic properties were conducted on the $(K_xSr_{1-x})CrO_z$, x = 0.10, 0.15 and 0.50 system; adjusted for different crystal sizes (polycrystalline, nano-crystalline and single crystals). For polycrystals, a solid-state reaction method was used (room temperature, T_R, up to 665 °C). Nano-crystals, a crushing technique with a mechanical mortar were processed at T_R. Single crystals were acquired, through fusion method starting from polycrystals pre-manufactured (665 °C) over 900 °C. For the methods described above, they all were worked at atmospheric pressure. Studying the thermodynamic in the crystals mixture were obtained by TGA (thermogravimetric analysis) and DSC (differential scanning calorimetry). Powder XRD (X-ray diffraction) in the manual grinding mixture a $SrCO_3$ solid solution, at T_R was detected. When increasing the temperature, a solid solution of $K_2Sr(CrO_4)_2$ is formed. By HRTEM (high-resolution transmission electronic microscope), crystal sizes 4-12 nm were seen. $(K_{0.50}Sr_{0.50})CrO_z$ composition presents a weak ferromagnetic and an insulating behavior.

Key words: Solid-state reaction, solid solutions, TGA-DSC, XRD, HRTEM, magnetic behavior.

Nomenclature

T_R	Room temperature
MM	Mechanical milling
MG	Manual grinding
C-AFM	C-type antiferromagnetic
AFM	Antiferromagnetic
XRD	X-ray diffraction
HRTEM	High-resolution transmission electronic microscope
TGA	Thermogravimetric analysis
DSC	Differential scanning calorimetry

1. Introduction

Previous studies have shown Pauli Paramagnetic behavior for $SrCrO_3$ compound, when a perovskite primitive cubic structure is present. At room temperature, the behavior displayed is not magnetic [1]. For a tetragonal structure it manifests C-AFM properties [2-5]. Although not much literature report, it is known about the magnetic properties of $SrCrO_3$ compound. Other studies have shown that the layered chromate from the perovskite structure type of $Sr_4Cr_3O_{10}$ compound demonstrates an isolating material as well as AFM, when produced at high pressure [6]. With the aforementioned in mind, the purpose of this investigation is to observe the change of physico-chemical properties when potassium ion is substituted in place of strontium ion. For example, producing a new $K_2Sr(CrO_4)_2$ solid solution at high temperature. Also the explanation of the formation of a $SrCO_3$ solid solution, observed by XRD, in the manual grinding mixture at room temperature. The electric and magnetic properties due to cationic-anionic substitution and hole formation were measured resulting an insulating and weak ferromagnetic material.

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2. Experimental Setup

2.1 Materials and Characterization

With stoichiometric calculation of reagents for preparing $(K_xSr_{1-x})CrO_z$, $x = 0.10$, 0.15 and 0.50 system, potassium dichromate KCr_2O_7, (ALDRICH, 99.99%) and strontium carbonate $SrCO_3$, (CERAC, 99.5%) were weighed in an analytical balance (OHAUS, ± 0.001 g). Previously the strontium carbonate was dried in the oven (Thermo Scientific, ± 4 °C) at 100 °C to make sure no water is present within the $SrCO_3$ compound. For polycrystals synthesis, the reagents were carried to MG (manual grinding) in an agatha mortar at P_{atm} (585 mmHg) for 30 min. To process the nano-crystals MM (mechanical milling) and a dry technique was used, the stoichiometric mixture reagents were crushed by MM for duration of 520 min in an agatha mechanical mortar (RM 100, Retsch model) at a speed of 370 RPM. Thermal analyzer (STD Q600, TA-instruments) was used, operating a standard alumina container, with the heating rate of 10 °C/min in air, over ranges T_{R} to 1,300 °C for TGA and DSC data. XRD was performed on a Bruker AXS D-8 advance diffractometer working with the Cu Kα wavelength ($\lambda = 1.5406$ Å), 40 KV and 30 mA. File sets were collected at room temperature, in a 2θ from 20° to 60° angular range, with a 0.020° step size and time per step of 2.4 s. For the HRTEM results, the equipment used was JEOL ARM F200, operating at 200 kV EDS Oxford Aztec. To measure the magnetic properties of the reagents and the final products, a SQUID MPMS3 model for ZFC-FC curves was used; the applied field was 100 Oe, range of measured temperatures 2-300 K. For the hysteresis curve, 5 K in maximum field 7 Tesla. A manual multimeter collected the electric data.

2.2 Synthesis of Polycrystals

For polycrystals synthesis, a solid-state reaction was used. Three samples were giving with $x = 0.10$, 0.15 and 0.50. Each reagents mixture was crushed MG in an agatha mortar for 30 min before, to carried out to the oven at 100 °C for 18 h. The MG samples were grinded every time heat treatment temperature was modified (T_{R} 665 °C) for a few days (1-108 d), at room pressure, with the aim of improving the contact area between the reagent's crystals. The temperature is increased every day after utilizing the MG technique, up to 665 °C each increase during a range approximately 13 h. The criterion used for the temperatures was obtained from the thermic results, they will be explained in detail in the results and discussions Section 3.

2.3 Synthesis of Nano-Crystals

The synthesis process to obtain nano-crystals was MM, a dry technique. The reagents for each sample, $x = 0.10$ and 0.50 were crushed by MM for a duration of 520 min in an agatha mechanical mortar. The MM samples were increased temperature at the same way those polycrystals, but only once in the mechanical mortar were crushed. The samples reaction started, from room temperature in the milling process at 24 °C up to 665 °C, for 84 d. The next crushing, MG technique was used each temperature increasing.

2.4 Single Crystal Synthesis.

The reagent's mixture forms MG and MM pre-manufacturing at 665 °C of $(K_xSr_{1-x})CrO_z$, $x =$ 0.10, 0.15 and 0.50 system, finally were reacted at 900 °C for several 50 min a semifusion product was obtained. Later were crushing manual in an agatha mortar and grow at 665 °C for 72 d, keep a solid solution with $x = 0.50$ by MM. Observing a mixture of compounds, presented in XRD data.

3. Experimental Results

3.1 Nano-Crystals Thermoanalysis Studies

To corroborate thermodynamic changes in nano-sizes crystals a TGA was made of $(K_{0.50}Sr_{0.50})CrO_z$ composition MM (Fig. 1). A small dent can be appreciated in the thermogram at 200 °C,

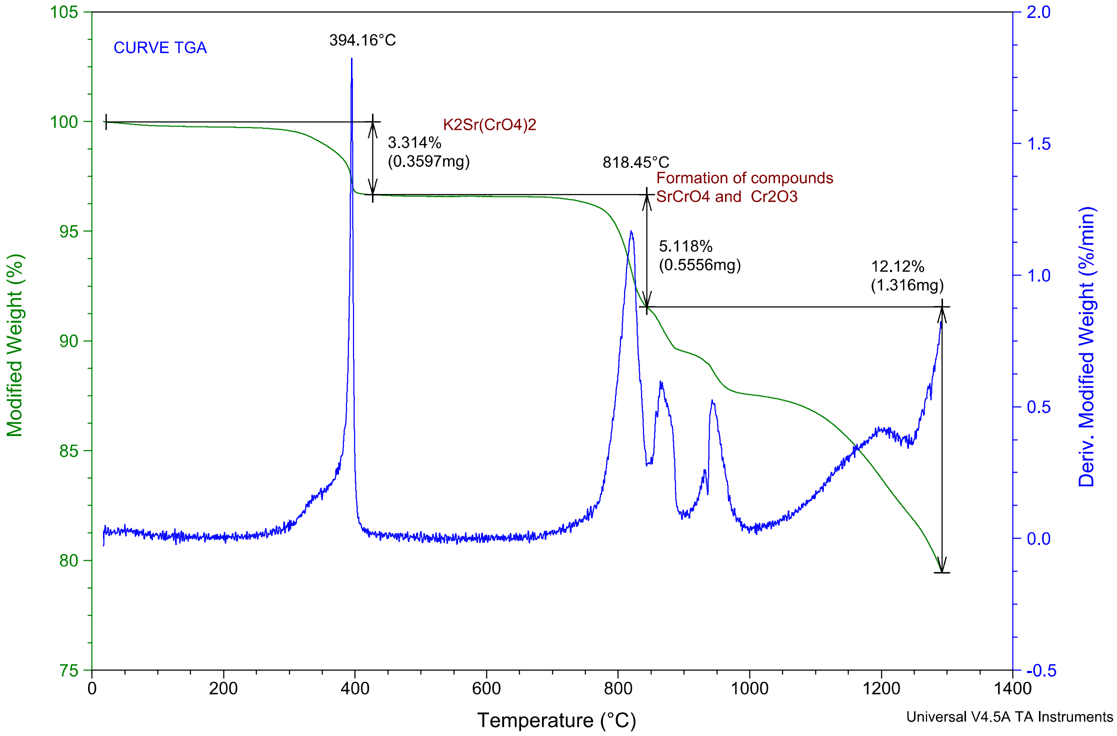

Fig. 1 DSC-TGA reagents mixture of $(K_{0.50}Sr_{0.50})CrO_z$ composition MM.

with this behavior we deduce a superficial dehydration. Mass loss of 3.314% at around 394.16 °C can be attributed to the intermolecular water loss, potassium dichromate decomposition and the reaction between the reagents successes. As observed in polycrystals, thermal analyzer (STD Q600, TA-Instruments) operating a standard alumina container, with the heating rate of 10 °C/min in air, over ranges T_R to 1,300 °C for TGA and DSC.

At temperatures above 800 °C the $K_2Sr[CrO_4]_2$ compound decomposes, due to the changes of the curves in the thermogram after the aforementioned temperature.

3.2 X-Ray Diffraction Studies

With powder XRD the purity of the reagents was observed, strontium carbonate (Fig. 2) (PDF-005-0418) and potassium dichromate (Fig. 3) (PDF-027-0380), at room conditions. With this information we can appreciated the formation of the solid solutions. Also the formation of $K_2Sr[CrO_4]_2$ compound (Fig. 4) (PDF-029-1047) MM. The mill sample of this analysis corresponds to a temperature of 665 °C/520 min and with x = 0.50. XRD was performed on a Bruker AXS D-8 advance diffractometer working with the Cu Kα1 wavelength (λ = 1.5406 Å), 40 KV and 30 mA. Data sets were collected at room temperature, in a 2θ from 20° to 60° angular range, with a 0.020° step size and time per step of 2.4 s.

Before starting a heat treatment reaction, of the reagent mixture at T_R (24 °C) was made. The sample for XRD analysis after 520 min was toke (Fig. 4). MM and MG prepared the samples. In MG at T_R, Fig. 5 a shift can be perceived, on $SrCO_3$ compound only in (113) and (241) planes and destructive (042) plane. See Table 1.

In $(K_{0.50}Sr_{0.50})CrO_z$ composition by MM at T_R a reaction is witnessed where $SrCO_3$, $K_2Sr[CrO_4]_2$, $KCr_2O_7/K_2O{\bullet}2CrO_3$ appear (PDF, 00-001-0676) and $SrCr_2O_4$ (PDF, 01-085-0370) with orthorhombic unit cell and S. G.: Pmmn (59) compounds (Fig. 6). The

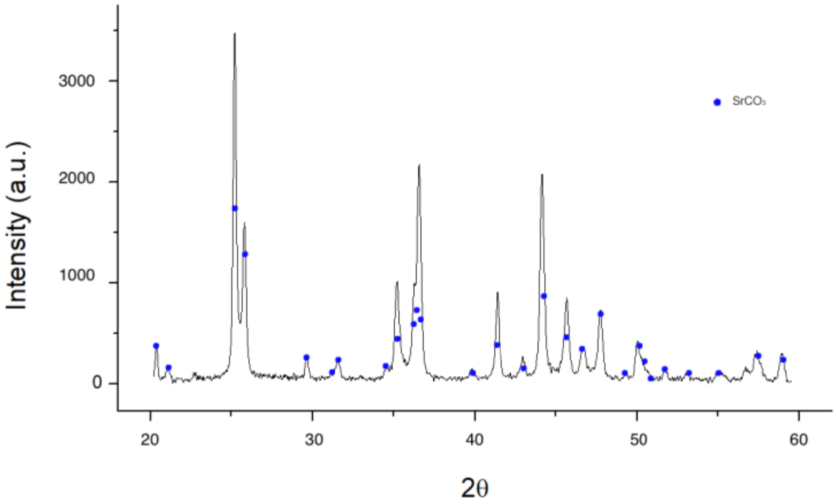

Fig. 2 XRD $SrCO_3$ compound.

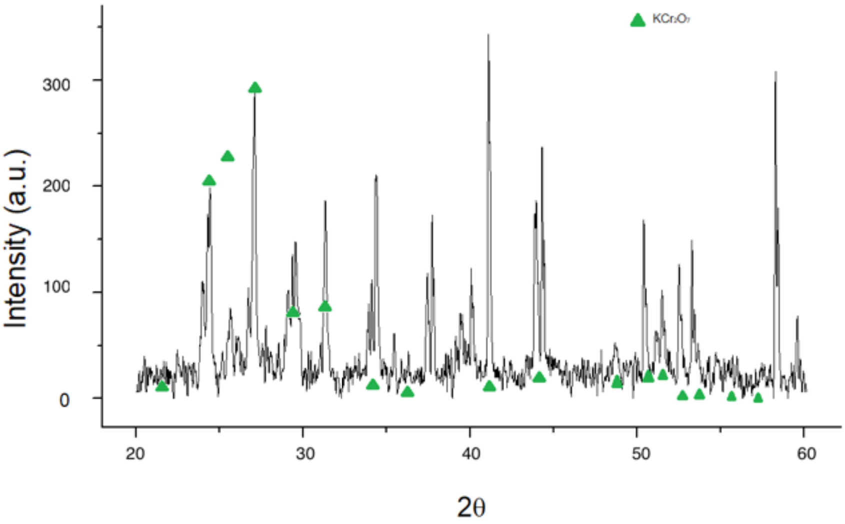

Fig. 3 XRD KCr_2O_7 compound.

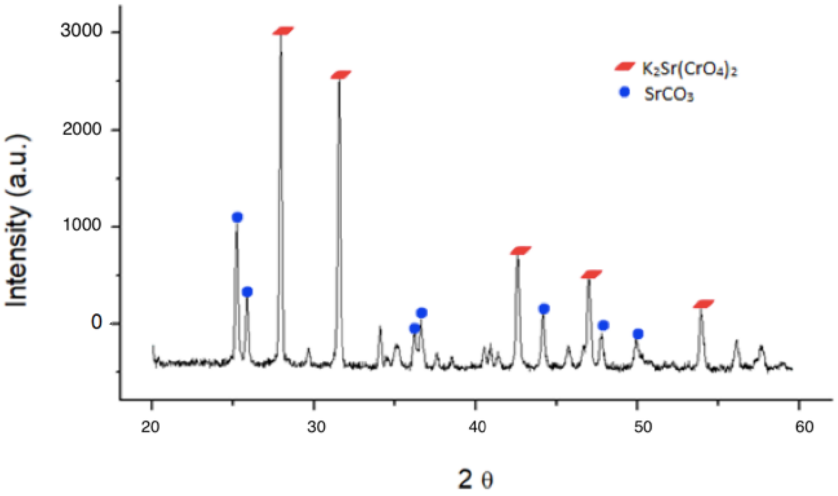

Fig. 4 XRD $(K_{0.50}Sr_{0.50})CrO_z$ composition at 665 °C MM 520 min.

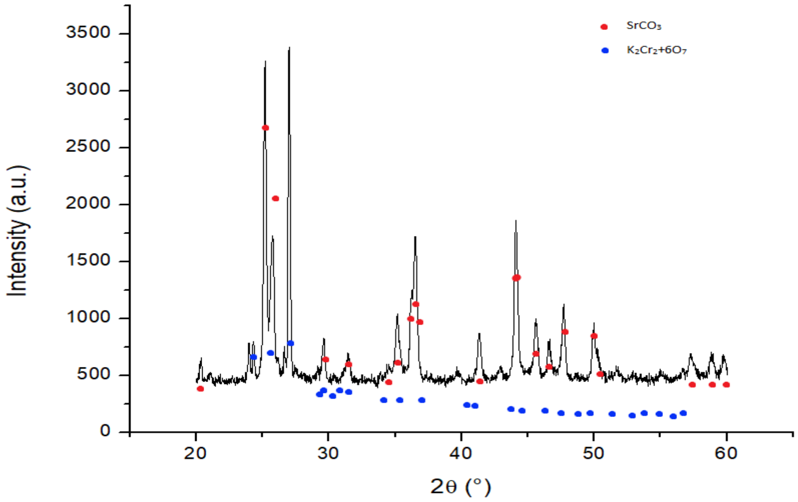

Fig. 5 XRD $(K_{0.50}Sr_{0.50})CrO_z$ composition at T_R MG.

Table 1 XRD data of $SrCO_3$ compound at room temperature.

$SrCO_3$ nano-cristal room temperature								MM	MG	
2θ teo	I	2θ exp	I	d	h	k	l	Δ2θ (exp-teo)	Δ2θ (exp-teo)	MG V. MM
20.319	14	20.344	32	4.3617	1	1	0	0.025	0.021	-0.004
25.172	100	25.177	100	3.5343	1	1	1	0.005	0.006	0.001
25.803	70	25.770	62	3.4544	0	2	1	-0.033	0.041	0.074
29.615	22	29.614	41	3.0141	0	0	2	-0.001	0.007	0.008
31.498	20	31.483	85	2.8371	0	1	2	-0.015	0.004	0.019
34. 522	12	34.036	35	2.6319	1	0	2	-0.497	0.019	0.516
35.108	23	35.136	36	2.5521	2	0	0	0.028	0.051	0.023
36.176	34	36.218	48	2.4783	1	1	2	0.042	0.088	0.046
36.527	40	36.520	50	2.4584	1	3	0	-0.007	0.007	0.014
41.323	16	41.337	33	2.1824	2	2	0	0.014	0.009	-0.005
44.083	50	44.110	55	2.0514	2	2	1	0.027	0.014	-0.013
45.643	26	45.612	39	1.9873	0	4	1	-0.031	0.039	0.07
46.563	21	46.590	37	1.9478	2	0	2	0.027	0.014	-0.013
47.694	35	47.695	44	1.9052	1	3	2	0.001	0.009	0.008
49.923	31	49.961	44	1.8240	1	1	3	0.038	0.021	-0.017
53.035	5	53.878	39	1.7002	0	4	2	0.843	Destructive	--
58.864	13	58.905	31	1.5665	2	4	1	0.041	0.014	-0.027
59.824	11	59.870	32	1.5436	1	5	0	0.046	0.072	0.026

weight of the pistil in MM produces the necessary energy to begin reaction without increasing the temperature where the system lowers its free energy by separating three compounds whose compositions are: $KCr_2O_7/K_2O{\bullet}2CrO_3$, $SrCr_2O_4$, and $K_2Sr[CrO_4]_2$.

Comparing Fig. 5 with Fig. 6 (T_R), in MG only reagents and in MM there are other compounds ($KCr_2O_7/K_2O{\bullet}2CrO_3$, $SrCr_2O_4$, and $K_2Sr[CrO_4]_2$). In both processes MG and MM, there are $SrCO_3$ solid solution formations.

3.3 Solid Solutions Study

XRD in Table 1, a shift in (021), (200), (112), (041) and (150) can be observed, show us a unit cell reduced. The (042) plane is a destructive reflection, deducing an empty hole. Adding temperature this plane appears. Table 4 shows the coordination number and ionic radium [6]; K^{1+} with 1.52 Å, with this information it can be possible to have an idea of substitution cations Sr^{2+} with 1.27 Å, and O^{2-} anion with 1.26 Å. It is viewed

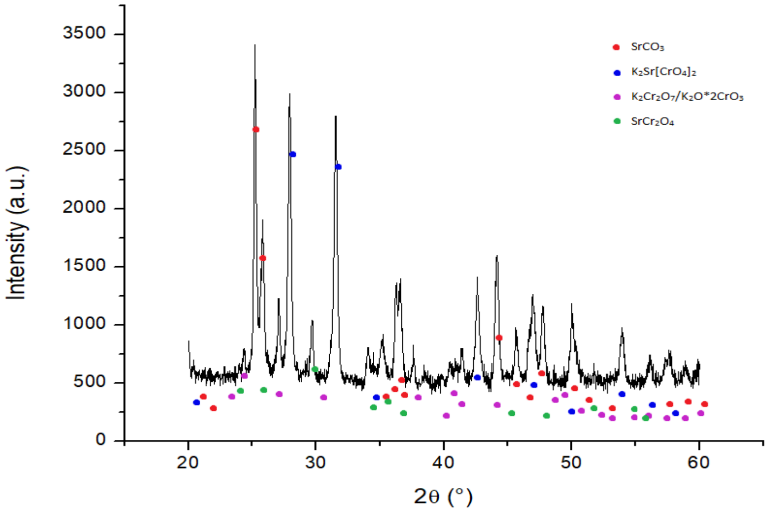

Fig. 6 XRD $(K_{0.50}Sr_{0.50})CrO_z$ composition at T_R MM 520 min.

a formation of solid solution at room temperature, then the mechanism in MG of $SrCO_3$ solid solution at T_R is complex [8], due to a K^{1+} cationic in Sr^{2+} ions substitution arrangement holes and O^{2-} anionic come in those empty sites conserved electroneutrality. It is necessary to perform radiation measurements with a synchrotron to elucidate the mechanism described above. At the moment we will not report that work.

MG at 665 °C in $(K_{0.50}Sr_{0.50})CrO_z$ composition it is found by XRD just $K_2Sr[CrO_4]_2$ compound solid solution. In Table 2: (110), (113), (202), (116), (125) and (300) have a displacement, deducing holes, and cationic-anionic substitutions.

For MM, $(K_xSr_{1-x})CrO_z$ with the variable x = 0.10, 0.15 and 0.50 system, a shift of some (*hkl*) in the $K_2Sr(CrO_4)_2$ crystal structure was observed. MM at T_R on $(K_{0.50}Sr_{0.50})CrO_z$ composition it had two solid solutions; $K_2Sr[CrO_4]_2$ and $SrCO_3$ compounds. In Table 5, $SrCO_3$ compound: (012), (102) and (042) are destructive planes and right side displacements occur in: (112), (241) and (150). Also for $K_2Sr[CrO_4]_2$ compound: (116) is destructive and displacements occurs in: (202) right side and (1010) left side. For right displacements, the cation substitution can be found in small sites and in left displacements in large sites.

Increasing the reaction temperature in MM, with respect to a TGA-DSC analysis (Fig. 1), a solid solution is formed in the $K_2Sr[CrO_4]_2$ compound (Table 2) and $SrCO_3$ (Table 3) remains unchanged. The strontium carbonate presence indicates that, it is not a saturated solid solution. At 450 °C in an $SrCO_3$ crystal structure, it changes from orthorhombic to rhombohedral. It is a rearrangement of cations and anions in the $SrCO_3$ compound.

MM at 665 °C in the $(K_{0.50}Sr_{0.50})CrOz$ composition, the following is detected: (113), (202), (116), (300) and (2010) displacement planes. Comparing this MM and MG at the same temperature it can be observed (113), (202), (116), and (300) planes highlight as shifts in the equivalent samples.

Comparing Fig. 4 MM 665 °C with Fig. 6 MM T_R in XRD of the $(K_{0.50}Sr_{0.50})CrO_z$ compound, a disappearance of $KCr_2O_7/K_2O•2CrO_3$ and $SrCr_2O_4$ can be appreciated, only the $K_2Sr[CrO_4]_2$ and $SrCO_3$ compounds remain. Due to the increase of temperature, the atoms are reorganized. Then in $SrCO_3$ compound (Fig. 4), it is assuming potassium ion substitution in the sites of strontium ion in the solid solution, because it is observed changes in some *hkl*.

When the x value in $(K_xSr_{1-x})CrO_z$ system increases, a greater percentage of the crystal structure of the ternary compound $K_2Sr[CrO_4]_2$ is obtained. It can be deduced that the saturation of the solid solution can be

Table 2 XRD data of $K_2Sr(CrO_4)_2$ compound.

$K_2Sr(CrO_4)_2$ nano-cristal 665 °C								MM	MG	
$2\theta_{\text{Theory}}$	I	$2\theta_{\text{exp}}$	I	d	h	k	l	$\Delta 2\theta_{\text{(exp-theory)}}$	$\Delta 2\theta_{\text{(exp-theory)}}$	MG v. MG
27.876	100	27.887	100	3.1967	0	1	5	0.011	0.019	0.008
31.475	90	31.502	88	2.8376	1	1	0	0.027	0.032	0.005
34.035	11	34.064	24	2.6298	1	1	3	0.029	0.037	0.008
37.507	5	37.550	17	2.3933	2	0	2	0.043	0.062	0.019
38.473	5	38.484	17	2.3373	0	0	9	0.011	0.023	0.012
40.472	6	40.487	19	2.2262	0	2	4	0.015	0.026	0.011
40.855	6	40.887	20	2.2053	1	1	6	0.032	0.033	0.001
42.570	35	42.568	43	2.1220	2	0	5	0.002	0.006	0.004
46.969	25	46.945	36	1.9339	1	0	10	0.024	0.029	0.005
53.881	20	53.906	29	1.6994	1	2	5	0.025	0.034	0.009
56.059	10	56.101	21	1.6380	3	0	0	0.042	0.035	-0.007
57.649	7	57.619	20	1.5980	0	2	10	0.030	0.024	-0.006

Table 3 XRD data of $SrCO_3$ compound at 665 °C.

$SrCO_3$ nano-cristal 665 °C								MM	MG	
$2\theta_{\text{Theory}}$	I	$2\theta_{\text{exp}}$	I	d	h	k	l	$\Delta 2\theta_{\text{(exp-theory)}}$	$\Delta 2\theta_{\text{(exp-theory)}}$	MM v. MG
20.319	14	20.323	16	4.3661	1	1	0	0.004	0.007	0.003
25.172	100	25.174	51	3.5348	1	1	1	0.002	0.004	0.002
25.803	70	25.817	32	3.4481	0	2	1	0.014	0.003	-0.011
29.615	22	29.602	19	3.0152	0	0	2	0.013	0.003	-0.01
31.498	20	31.502	88	2.8376	0	1	2	0.004	0.009	0.005
34.522	12	34.467	16	2.6000	1	0	2	0.066	0.002	-0.064
35.108	23	35.086	20	2.5555	2	0	0	0.022	0.023	0.001
36.176	34	36.198	22	2.4795	1	1	2	0.022	0.028	0.006
36.527	40	36.575	26	2.4548	1	3	0	0.048	0.039	-0.009
41.323	16	41.360	18	2.1812	2	2	0	0.037	0.034	-0.003
44.083	50	44.129	27	2.0505	2	2	1	0.046	0.028	-0.018
45.643	26	45.689	19	1.9841	0	4	1	0.046	0.009	-0.037
46.563	21	46.621	19	1.9466	2	0	2	0.058	0.077	0.019
47.694	35	47.717	22	1.9044	1	3	2	0.023	0.012	-0.011
49.923	31	49.905	21	1.8259	1	1	3	0.018	0.004	-0.014
53.035	5	53.906	29	1.6994	0	4	2	0.871	0.005	-0.866
58.864	13	58.901	15	1.5667	2	4	1	0.037	0.046	0.009
59.824	11	59.947	15	1.5418	1	5	0	0.123	0.004	-0.119

Table 4 Cations and anion coordination number.

COORD	[4] Å	[6] Å	[8] Å	[12] Å
ELEM				
O^{2-}	1.24	1.26	1.28	1.21
Cr^{3+}	--	0.755	--	--
Cr^{4+}	0.58	0.69	--	--
Cr^{5+}	0.49	--	0.71	--
Cr^{6+}	0.44	--	--	--
Sr^{2+}	--	1.27	1.39	1.54
K^{1+}	--	1.52	1.65	1.74

Table 5 XRD data of MM $SrCO_3$ & $K_2Sr[CrO_4]_2$ compound at T_R.

$SrCO_3$ & $K_2Sr[CrO_4]_2$ nano-cristal T_R								Displacement
2θ teo	I	2θ exp	I	d	h	k	l	Δ2θ (exp-teo)
$SrCO_3$	$K_2Sr[CrO_4]_2$							
20.319	14	20.344	32	4.3617	1	1	0	0.025
25.172	100	25.177	100	3.5343	1	1	1	0.005
25.803	70	25.770	62	3.4544	0	2	1	-0.033
27.876	100	27.880	90	3.1975	0	1	5	0.004
29.615	22	29.614	41	3.0141	0	0	2	-0.001
31.475	90	31.483	85	2.8393	1	1	0	0.008
31.498	20	--	--	--	0	1	2	--
34.035	11	34.036	35	2.6319	1	1	3	0.001
34.522	12	--	--	--	1	0	2	--
35.108	23	35.136	36	2.5521	2	0	0	0.028
36.176	34	36.218	48	2.4783	1	1	2	0.042
36.527	40	36.520	50	2.4584	1	3	0	-0.007
37.507	5	37.576	35	2.3917	2	0	2	0.069
38.473	5	38.504	29	2.3362	0	0	9	0.031
40.472	6	40.443	32	2.2285	0	2	4	-0.029
40.855	6	--	--	--	1	1	6	--
41.323	16	41.337	33	2.1824	2	2	0	0.014
42.570	35	42.540	43	2.1234	2	0	5	-0.030
44.083	50	44.110	55	2.0514	2	2	1	0.027
45.643	26	45.612	39	1.9873	0	4	1	-0.031
46.563	21	46.590	37	1.9478	2	0	2	0.027
46.969	25	46.893	46	1.9359	1	0	10	-0.076
47.694	35	47.695	44	1.9052	1	3	2	0.001
49.923	31	49.961	44	1.824	1	1	3	0.038
53.035	5	--	--	--	0	4	2	--
53.881	20	53.878	39	1.7002	0	4	2	-0.003
56.059	10	56.086	33	1.6385	3	0	0	0.027
57.649	7	57.629	33	1.5982	0	2	10	-0.020
58.864	13	58.905	31	1.5665	2	4	1	0.041
59.824	11	59.870	32	1.5436	1	5	0	0.046

found on the other side of the system with $x > 0.50$, which is currently under study.

For the monocrystals the fusion method was used, at 900 °C the material presents decomposition and new compounds were produced. In the $(K_{0.50}Sr_{0.50})CrO_z$ composition at 900 °C for MM; $SrSO_4$, K_2SO_4, $SrCO_3$, $K_2Sr[CrO_4]_2$, Cr_2O_3, $SrCrO_4$ and $K_2Sr[SrO_4]_2$ were found (Fig. 7).

The crucibles used for the fusion method were washed with H_2SO_4, finding sulphur compounds in the new mixture. It remained the same rhombohedra crystal structure for $K_2Sr[CrO_4]_2$ and orthorhombic crystal structure for $SrCrO_4$ and Cr_2O_3. It is learned that the material must be washed for a longer time when acid is used to remove residues in the crucibles.

3.4 High-Resolution Transmission Electronic Microscope Studies

Fig. 8 shows a cluster of the $K_2Sr[CrO_4]_2$ compound in the $(K_{0.50}Sr_{0.50})CrO_z$ composition with MM during 520 min at 665 °C. Described in 2.1 synthesis section. Moreover, in Fig. 9 it is possible to see polycrystals and nano-crystals. Fig. 10 is a single nano-crystal with (015) since 2.36-3.71 Å.

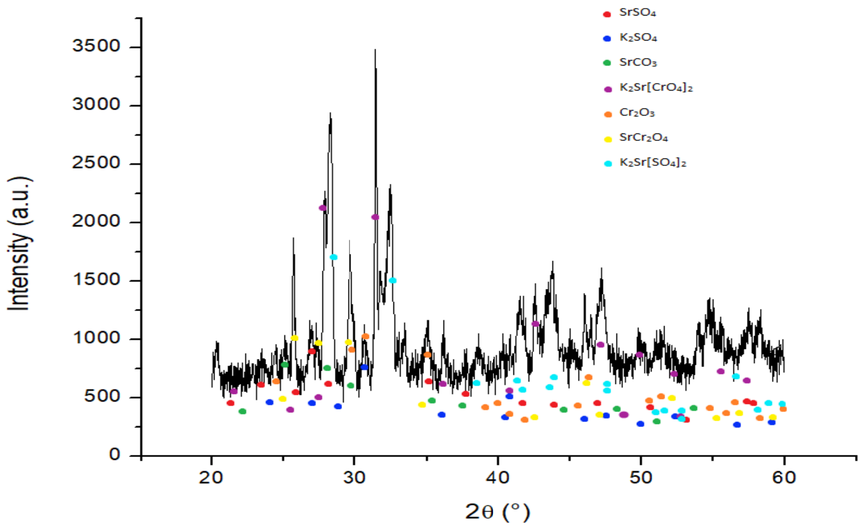

Fig. 7 XRD $(K_{0.50}Sr_{0.50})CrO_z$ composition at 900 °C MM 520 min.

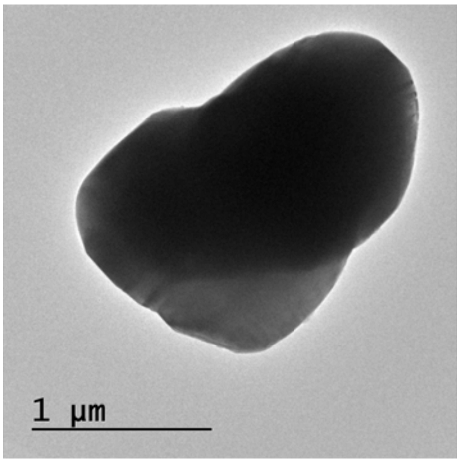

Fig. 8 HRTEM $(K_{0.50}Sr_{0.50})CrO_z$ composition at 665 °C MG 520 min.

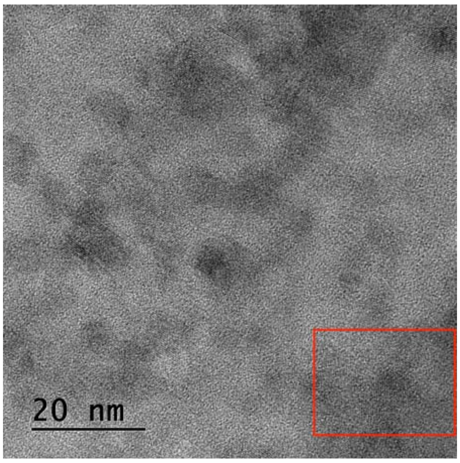

Fig. 9 HRTEM $(K_{0.50}Sr_{0.50})CrO_z$ composition at 665 °C MM 520 min.

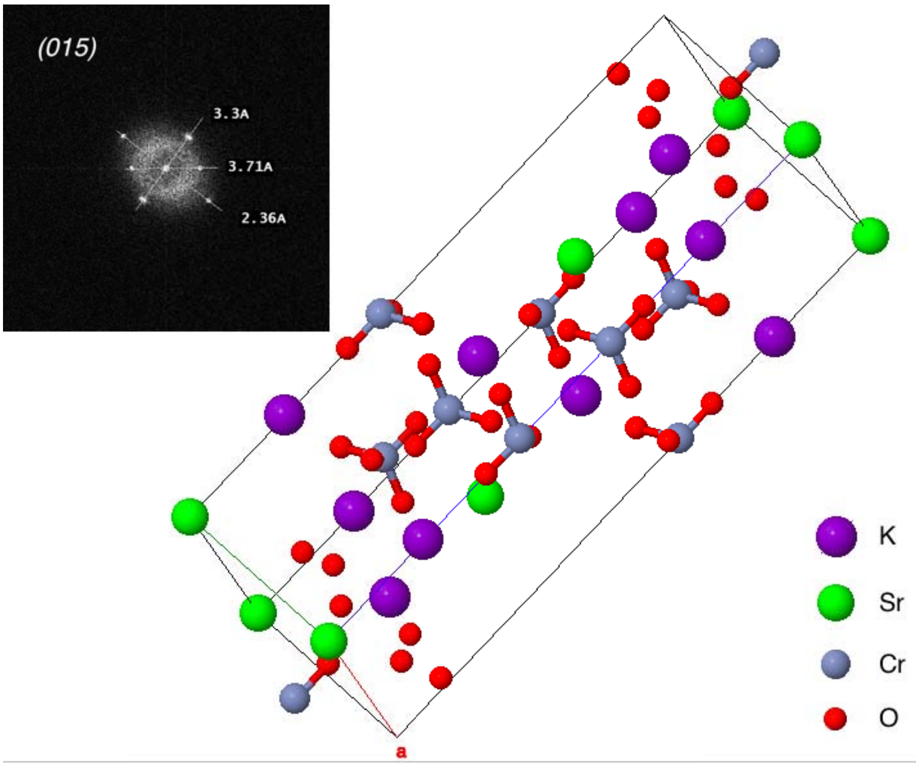

Fig. 10 HRTEM of a single crystal with (015) since 2.36-3.71 Å in $(K_{0.50}Sr_{0.50})CrO_z$ composition at 665 °C MM 520 min. Unit cell [7].

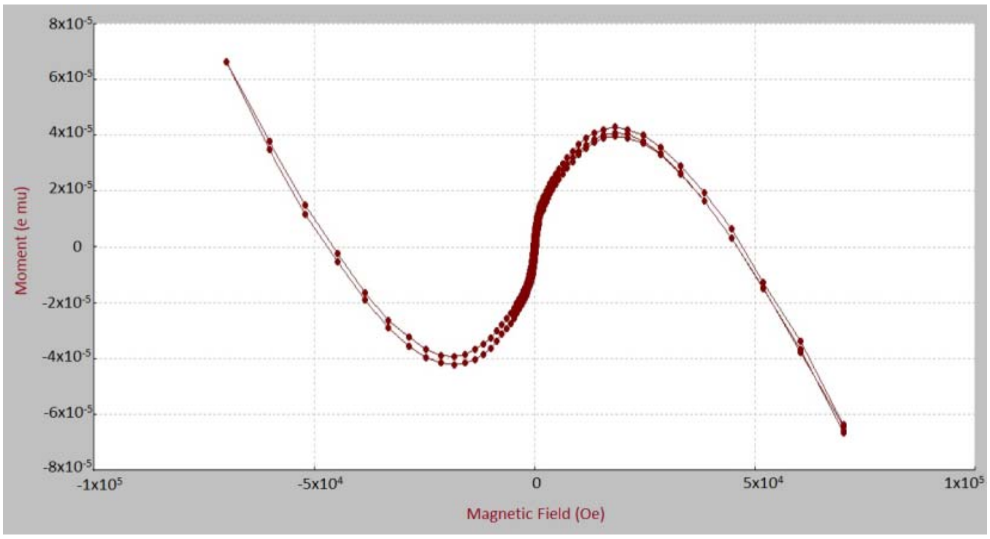

Fig. 11 $(K_{0.50}Sr_{0.50})CrO_z$ composition MM 665 °C hysteresis.

3.5 Magnetic Studies

In Fig. 11 in the $(K_{0.50}Sr_{0.50})CrO_z$ compound, we can observe in -2 T to 2 T range hysteresis, this compound is a weak ferromagnet. From 2 T a downward trending curve as a diamagnetic behavior can be seen, because the material opposite a high field is applied. The symmetry repeats in the same fashion on the other side (-2 T).

3.6 Electric Measurements Studies

In the $(K_{0.50}Sr_{0.50})CrO_z$ MM compound at 665 °C, was measured at T_R with a voltmeter resulting high resistance as insulating (Fig. 11).

4. Conclusions

For x = 0.10, 0.15 and 0.50 values in the $(K_xSr_{1-x})CrO_z$ system, different solid solutions depending on MM, MG and/or T_R—heat treatment reaction conditions are detected. The x = 0.50 at 665 °C in MM was identified in a sample containing in a majority of the $K_2Sr[CrO_4]_2$ compound. Using the XRD method, it could find in $K_2Sr[CrO_4]_2$ compound their rhombohedral crystal structure. In their nano-crysatlline form with 4-12 nm range, $(K_{0.50}Sr_{0.50})CrO_z$ composition is a weak ferromagnetic and isolate material.

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