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Structure Study of Mn Doped in $SrTiO_3$ by X-ray Diffraction

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Abstract - Mn:$SrTiO_3$ monocrystal was prepared from these compounds MnO_2, TiO_2, $SrCO_3$ by equivalent weight at 2.52g.Mn doped to sample by 60% mole. The sample under studied had size of 3mm thickness, and 12mm diameter at room temperature T=300K=27^{0}C which has annealing temperature equal to1223K= 950^{0}C for 24 h. The structure studied for this crystal by XRD way. The lattice constant for Mn: $SrTiO_3$ crystal was calculated where a=3.269^{o}A. It structure was cubic type ($P\ m\bar{3}m$). The grain size of this crystal investigated by (SEM), it was 5μm at 5000 time magnified.

Keywords**:** Spectrum, grain, Structure factor, Atomic scattering factor, Lattice, Annealing.

1. Introduction

$SrTiO_3$ (STO) crystal is one of perovskites materials ABO_3. It has along range of applications as used in transistor, receiver, non-vital memory devices and so on [1]. It is semiconductor materials but it becomes superconductor, when doped Mn or any other transition metal ions in it [2, 1]. It depended on that, a lot of physics properties developed and gave the best results as conductivity and magnetic properties [2, 3].

It is worth to mention that, most the ABO_3 crystals nearly related in preparation method. Several preparation technique developed to produced Mn: $SrTiO_3$ nanoparticales including sol-gel method, organic precursor method [4], powder samples method[5], and so on.

In this paper, the monocrystal sample of Mn:$SrTiO_3$ was prepared by powder samples where Mn doped in $SrTiO_3$ crystal. Then used x- ray diffraction(XRD) to investigated the structure, where it was the first study to investigate the structure for that crystal by (XRD) and low resolution device with maximum I =1mA and voltage V=25kV, also the grain size founded by scanning electron microscopic (SEM).

2. Material and methods

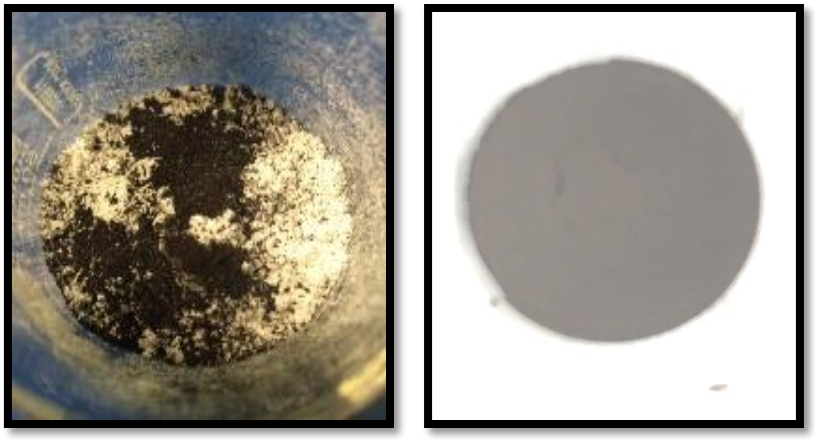
2.1. Preparation of Mn:$SrTiO_3$ monocrystal

The Mn: $SrTiO_3$ monocrystal prepared from these compounds as powder MnO_2, Tio_2, $SrCo_3$ [2, 5, 6] with pure 70%, 99%, 98% respectively. These compound added by equivalents ratio [5] at 2.522g, so the ratio of doped Mn in $SrTiO_3$ was equal to 60% mole. These powders were mixed by magnetic stirrer for one hour to be the content of the flask homogenous, and then it was soaked by De-Ionzed (DI) water for 20-25min [7, 8]. HCL-HNO_3 (3:1 ml) acidic solution was added and mixed for12 min [9, 8]. The sample was annealing at 1223K (heated in furnace at atmosphere to 1223K= 950^{0}C for one hour [7, 10, 2], then was could to 24hours). It was grinded and pressed [2, 11] in thickness 3mm and 12mm diameter. Then the monocrystal was formed as in fig (1).

2.2. Study Methods

(XRD) way was used to study the structure of this monocrystal by low resolution device (X-ray apparatus 55481) with Molybdenum (MO) Anode, maximum current intensity was I= 1mA, and voltage equal to V=20kV [12].

(SEM) was used to find grain size of this monocrystal.



A B

Fig. 1: A) powder compound mixing, B) Mn:$SrTiO_3$ monocrystal in 3mm thick and 12mm diameter.

3. Results and dissections

3.1. Structure Study by XRD

The best spectrum of this monocrystal was gotten by several steps: first step was searched range of voltage V= 10, 15, 20, 25 kV and fixed the current intensity at I=1mA, as in Fig(2) . From this figure was cleared that at high voltage the deformation in the shape of spectra lines duplicated in some parts of spectra more than the others. it was boor spectra. At low voltage, the number of reflection planes in crystal decreased because there were several electrons couldn't gain enough energy to arrive to the cathode, so the x-ray was very weak [12]. From fig (2) the best spectrum of this monocrystal was cleared at voltage V=20kV, and number of reflection planes (lines) agree with others [6].

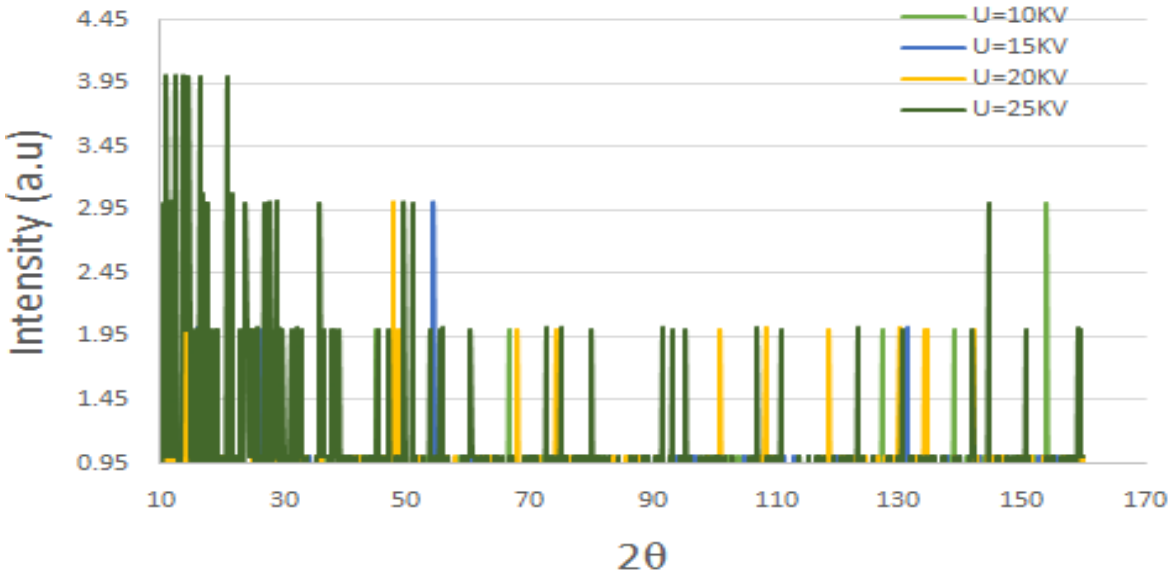


Fig. 2: spectrum lines of Mn:$SrTiO_3$ with variable voltage.

The second step to get the best spectrum to that crystal was gotten by searching for range of current intensity I= 0.5, 0.7, 0.75, 1 mA and fixed the voltage at V=20kV as in fig (3). At low current intensity, the numbers of reflection planes decreased. So the best spectrum lines were at I= 1mA which agreed with number of lines of publishing papers [11, 13].

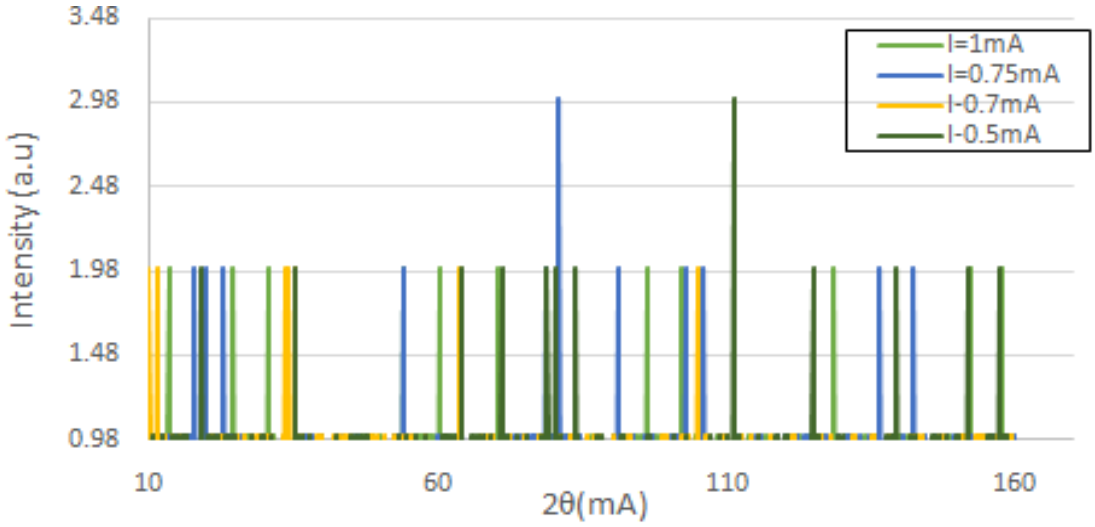


Fig. 3: spectrum lines of Mn:$SrTiO_3$ with variable current intensity.

After searching was found the best spectrum for Mn:$SrTiO_3$ was at current intensity I= 1mA , and voltage V=20kV with T=300K as in fig(4). It consisted of eleven reflection planes that was agree with others [6,13].

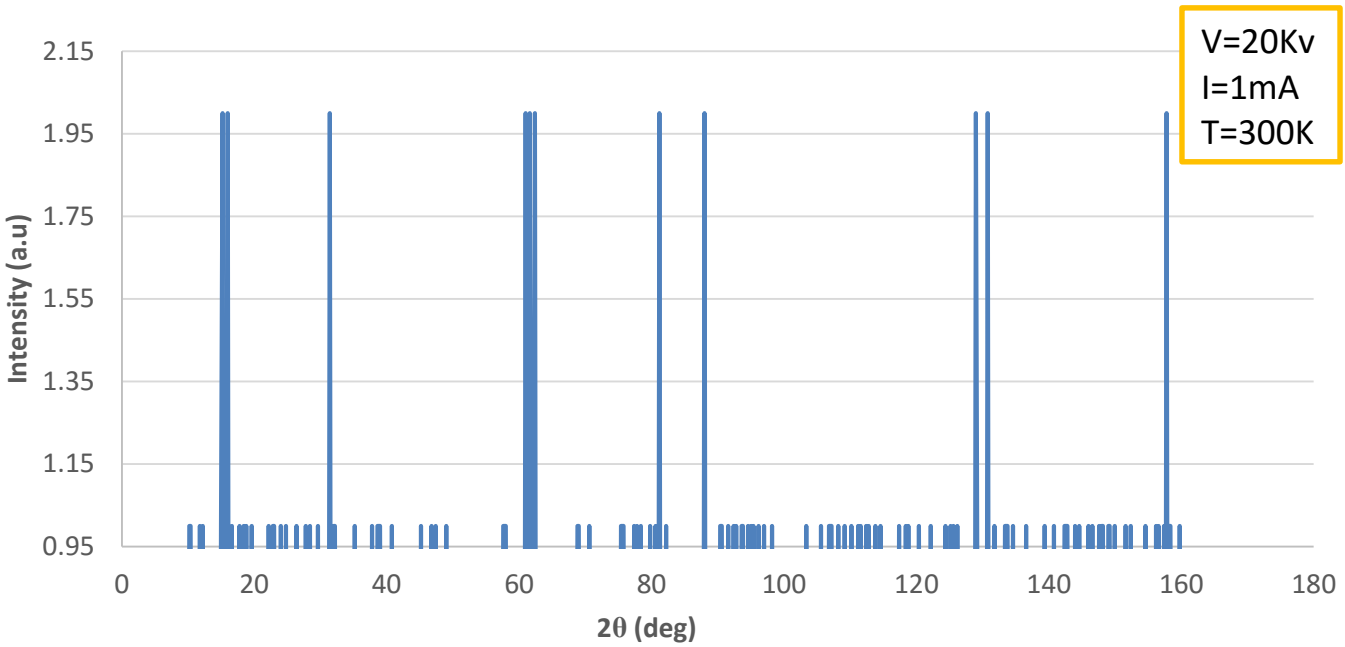


Fig. 4: Best spectrum lines of Mn:$SrTiO_3$ crystal.

The wavelength of x-ray was calculated from eqn. (1)

$$\lambda = \frac{hc}{eV} \tag{1}$$

$$\lambda = \frac{1.24 \times 10^{-6}}{V} \tag{2}$$

$$\lambda = 0.62\dot{A} \tag{3}$$

The lattice constant (a) calculated from the slope in fig (5) and Brag's law equ. (4), where it equal to distance (d) between planes in crystals as in the following equations [14]:

$$n\lambda = 2dsin\theta \quad (4)$$

$$a = slpe/2 \quad (5)$$

$$a = 3.269\dot{A}^{\pm}(0.008\%) \quad (6)$$

The lattice constant value was nearly to the value of others [5, 15, 6]. The reason of the small amount differences was the ratio of Mn doped in STO was very high, so it was heavy doped. If the doping in the crystal increased, the value of lattice constant decreased and vice versa [15].

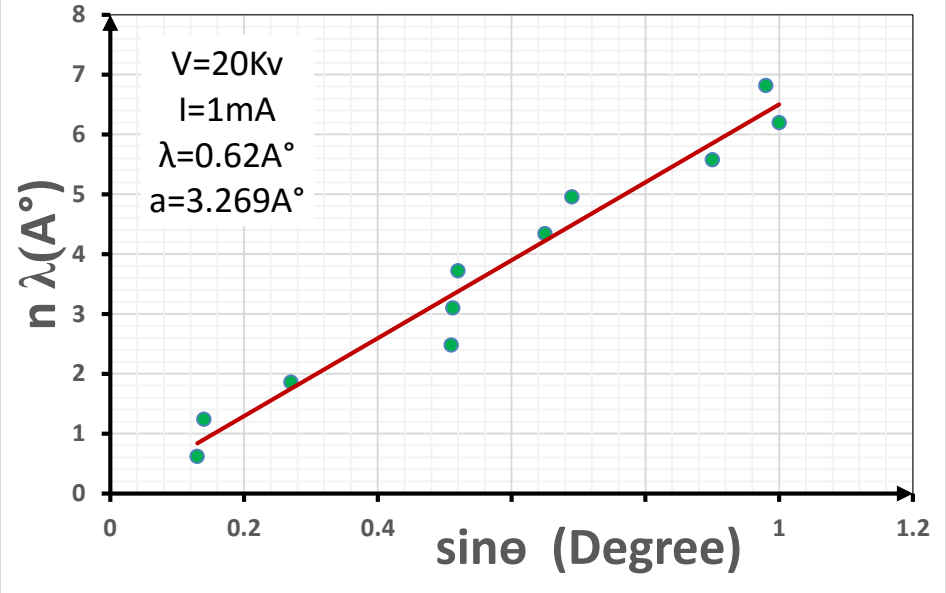


Fig. 5: The relation between the wavelength of different reflection order (nλ) and the diffraction angle (θ).

The structure factor was calculated from the atomic scattering factor (f), and the spectrum intensity (R) as in equ. (7), fig (6) [14].

$$R_{h,k,l} = \left|f^2_{h,k,l}\right| \quad (7)$$

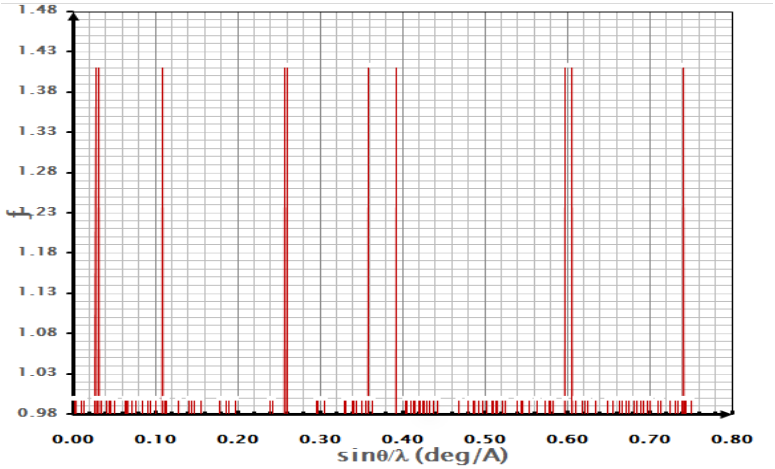


Fig. 6: Atomic scattering factor.

The atomic scattering factor was constant because the crystal had magnetic properties. It wasn't affected by incidence angle of x-ray. The atomic scattering factor was valuable affected by neutron diffraction. Although, the structure factor (S) could be calculated from this value of atomic scattering factor. From equ. (8) and fig (6) could be calculated the structure value. It had value equals to zero as in equ. (10), [16].

$$S = \sum_n f_n e^{2\pi(hu+kv+lw)} \tag{8}$$

$$S = \frac{(f_1 - f_2)}{|f_1 - f_2|} + \Delta f \tag{9}$$

$$S = 0 \tag{10}$$

Because the structure factor was zero, so this crystal was cubic from type ($P\ m\bar{3}m$) as in fig (7), [16]. This result was agreed with others [1]. Where Mn^{2+} substituted with Ti^{2+} and Mn^{4+} substituted with Sr^{4}+ [1, 6].

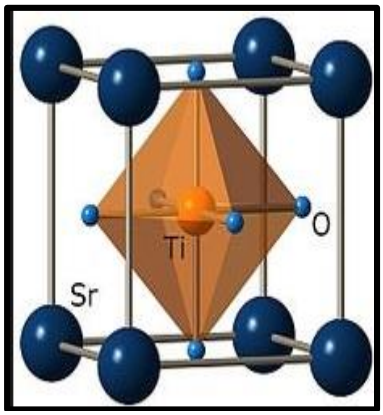


Fig. 7: structure of Mn:$SrTiO_3$ crystal ($P\ m\bar{3}m$).

3.2. Grain Size by SEM Technique

The grain size of this monocrystal was calculated by (SEM) as in fig (8) with magnified was 5000 times. The grain size crystal was 5µm. this value was agreed with others [4]. The grain size of crystal depended on the doping ratio of crystal. If the doping ratio of crystal increased, the grain size will be decreased [15,6]. The table (1) was given brief summary to the results.

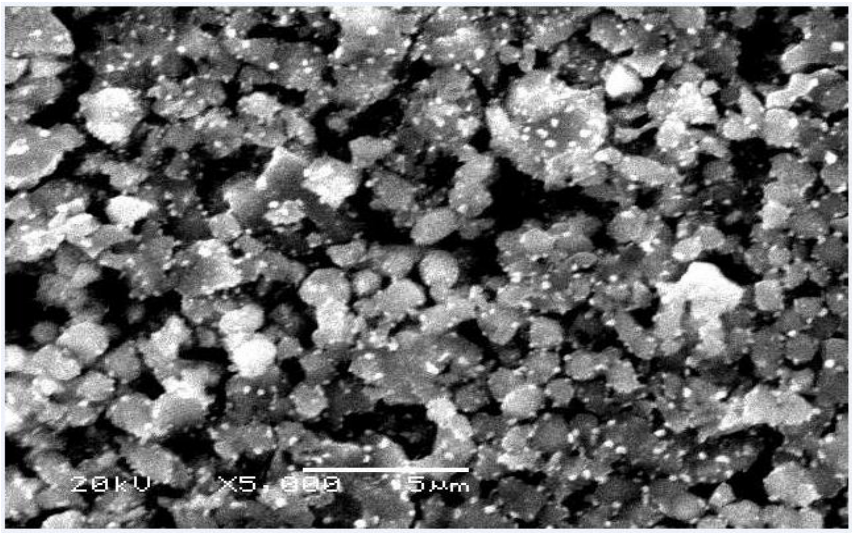


Fig. 8: Grain size of Mn:$SrTiO_3$ crystal.

Table 1: brief summary of results.

wavelength	**λ=0.62^{0}A**
Mn ratio in crystal	**60% mole**
crystal size	**Thicknes 3mm diameter 12mm**
Annealing temperature	**1223K= 950^{0}C**
Crystal temperature	**300K= 27^{0}C**
Lattice constant(a)	**a = 3.269^{0}A**
crystal structure type	($P\ m\bar{3}m$) **cubic**
Crystal Grain size	**5µm**

4. Conclusion

This was the first structure study of Mn:$SrTiO_3$ had 60% mole doped of Mn in STO and investigated by XRD way. Where get the same result of neutron diffraction study for Mn doped in STO. The structure of this crystal was cubic type ($P\ m\bar{3}m$), and the lattice constant was 3.269^{0}A. The grain size was 5µm for magnified 5000 times. It was hard to make the crystal because the literature didn't clear enough and there were different ways to doping. Also, the crystal made at high temperature which requested special Furness. The low resolution x-ray device required special skills to adapt.

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