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Microhardness measurements on Ca²⁺ doped triglycine sulphate crystal

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ABSTRACT

Pure and calcium²⁺ doped triglycine sulphate crystals were grown from aqueous solution by slow evaporation technique, Vicker's microhardness measurement were done on all the grown crystals. The difference in microhardness values vary non-linearly with calcium concentration.

Keywords: TGS; Crystal growth; Doping; Rare earth; Micro hardness

INTRODUCTION

Crystals of Triglycine Sulphate (TGS) is a well known ferroelectric materials, find wide application as room temperature IR detectors [1]. TGS is an order-disorder type ferroelectric with a transition from ferroelectric phase at 49° C, having high pyroelectric coefficient and low dielectric constant values[2]. TGS crystals have a tendency to polarization reversal. In order to overcome this difficulty, the dopant has been added to the pure TGS crystals. Also, Armingston et al [3] discussed two methods of improving hardness (i) solid solution hardening (ii) impurity hardening. In this views, in the present study Calcium has been added to the pure TGS crystals to overcome the above .

Chin et al [4] study effect of divalent impurity on the hardness of sodium and potassium halides . This paper discussed about the hardness of calcium doped TGS crystals.

MATERIALS AND METHODS

TGS salts were synthesized from the following reaction

 $3(NH_2CH_2COOH) + H_2SO_4 \rightarrow (NH_2CH_2COOH)_3(H_2SO_4)$

AnalaR Grade Glycine and Sulphuric acid were used for the synthesize of TGS salt, after successive recrystallization processes the purified salt were used for the Preparation of the solution. Calcium carbonate was added in the ratio viz. 1:0.000, 1:0.002, 1:0.004, 1:0.006, 1:0.008 and 1:0.010 to the TGS solution and saturated at 45° C.

Growth was initiated by a temperature reduction of the solution and slow cooling was employed. After a few days of growth the crystals were harvested after a typical growth period of 10 days.

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The dopant concentration in the grown crystals were determined from Atomic

Absorption Spectrum data. The Vicker's micro hardness measurement were carried out using ZWICK 3212 hardness tester.

The microhardness number can be determined from using the formula

 $Hv = 1.8544P/d^2 \text{ kg mm}^{-2}$

Where, $P \rightarrow$ applied load in kg, $d \rightarrow$ average diagonal length of the Vicker's impression in mm after unloading.

The Meyer's work hardening co-efficient m can be determined by plotting log p vs log d, the variation of log p vs log d for the sample 1:0.002 is shown in figure 2. as an illustration.

The slope of best linear fit graph gives "m" value

RESULTS AND DISCUSSION

The values of micro hardness for TGS crystals doped with different mole fraction of Ca^{2+} are presented in the table1. The increment in hardness numbers (ΔHv) due to doping is calculated as difference between hardness number of the given doped crystals and the hardness of the pure TGS crystals. The values of (ΔHv) for all the doped crystals are also presented in table1. for the loads 25gm, 50gm and 100gm. In figure 1. the increment in the values of hardness (ΔHv) for Ca^{2+} doped TGS crystals are plotted against the concentration of the dopant. It is observed that the increment in the hardness varies non-linearly with the concentration of the dopant. Makin et al [5] derive a law according to which the hardening is proportional to the concentration, C as $C^{1/3}$. Fleischer [6] and Gilman [7] adopting slightly different treatment have shown that hardening should be proportional to $C^{1/2}$.

In order to determine the value of n, for the present system, the data is fitted by the least square procedure to the following equation

$\Delta Hv = kc^n$

The values of k and n for the three loads 25gm, 50gm and 100gm are provided in the table2. It is observed that the values of n are close to 0.5 for the system with load 25gm and 50gm, which is close to the value predicted by Fleischer [6] and Gilman [7].

Meyer's work hardening co-efficient "m" values for different composition are shown in table 3. According to Onitsch[8] and Hanneman [9] the m values fall below 1.6 for hard materials and more than 1.6 for soft materials. The values obtained in the present study imply that the grown crystals belong to hard materials category.

System	Calcium concentration(ppm)	Hardness Number			ΔHv		
		25 gm	50 gm	100 gm	25 gm	50 gm	100 gm
PureTGS	0	18.75	27.85	42.35	0	0	0
1:0.002	9.9132	27.2	43.35	78.6	8.45	15.5	36.45
1:0.004	10.645	38	54.4	80.8	19.25	26.5	38.45
1:0.006	17.396	48.8	62.8	87.4	30.05	34.95	45.05
1:0.008	26.145	52.65	66.4	96.85	33.9	38.55	51.7
1:0.010	85.5161	56.35	72.15	94.05	37.6	44.25	54.5

Table 1. values of hardness number	r (Hv kg mm ⁻²), Δ Hv along with system
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Figure1. Concentration vs Difference in micro hardness



Figure 2.Values of log p vs log d

Table 2. values of k and n

load	k	n
25 gm	6.6414	0.5917
50 gm	5.941	0.4276
100 gm	5.0452	0.2099

Table3. values of work hardening co-efficient (m)

system	work hardening co-efficient (m)
Pure TGS	0.221
1:0.002	0.136
1:0.004	0.238
1:0.006	0.251
1:0.008	0.281
1:0.010	0.315

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CONCLUSION

The micro hardness value varies non-linearly with concentration and the n values agree with Fleischer and Gilman. All the grown crystals belong to hard category.

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