GROWTH OF PURE AND DOPED TGS CRYSTALS

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ABSTRACT

Ferroelectric single crystals of pure TGS and TGS doped with copper are grown by solution method. A comparative study of the growth and perfection of the crystals is reported. Microtopography of the cleavage plane (010) is studied. It is observed from the dislocation density measurements, microtopography and macrostudy of the crystals that the copper doped TGS crystals are more perfect.

PURE triglycine sulfate $(NH_2CH_2COOH)_3 . H_2SO$ crystals (TGS) are recently used in infrared detectors, multielement arrays and for thermal imaging in the pyroelectric Vidicon[1–3]. Pure and doped TGS crystals have been grown to study their defect structure, microhardness and ferroelectric properties as a function of composition. This note reports the study of growth of pure TGS crystals and TGS crystals doped with copper. A comparative study of perfectness and growth rate is also discussed.

TGS was prepared by mixing A.R. glycine and A.R. H_2SO_4 in 3:1 molar proportion[4–5]. Saturated solution of TGS was prepared in distilled water at 27° C and the solution was evaporated at the same temperature for the preparation of seeds when rod type and plate type seeds were obtained. These seeds were well defined and transparent. It has been found that perfectness and growth rate are higher when rod type seeds are used for crystal growth. In the present investigation rod type seeds of mass about 0·1 g and dimensions (0·7 × 0·4 × 0·1 cm) were used. Crystals were grown by solution method, care being taken to avoid temperature fluctuations and vibrations. Well defined crystals of mass about 24 g and dimensions (6·4 × 2·0 × 1·8 cm) have been grown in about 39 days (Fig. 1). The photograph clearly shows that the crystal is not perfectly transparent. In almost all the cases prominent habit faces are (100), (010), (011) and (111).

Crystals were cleaved along (010) plane at room temperature. Figs. 2 (*a*) and 2 (*b*) show one to one correspondence of the features. Roughness of the cleavage plane is clearly visible in the photomicrograph.

Freshly cleaved specimens were etched in a dislocation etchant developed by the present authors (1 part distilled water + 4 parts A.R. acetic acid). The etchant reveals the dislocations (Fig. 3) on the cleavage plane by producing rectangular point-bottomed pits. The average dislocation density in the specimens was found to be ~10^4 per cm^2. The perfectness of the crystals was also checked by X-ray method.

FIG. 1. Pure TGS single crystal.

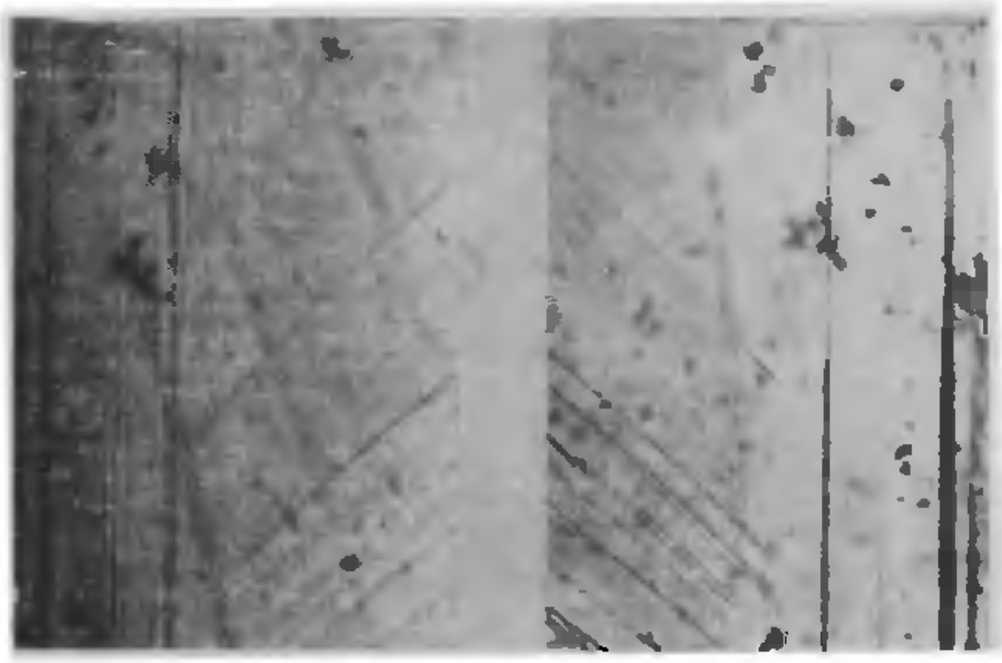

FIG. 2 *a*, *b*. Oppositely matched cleavage faces of pure TGS crystal (× 120).

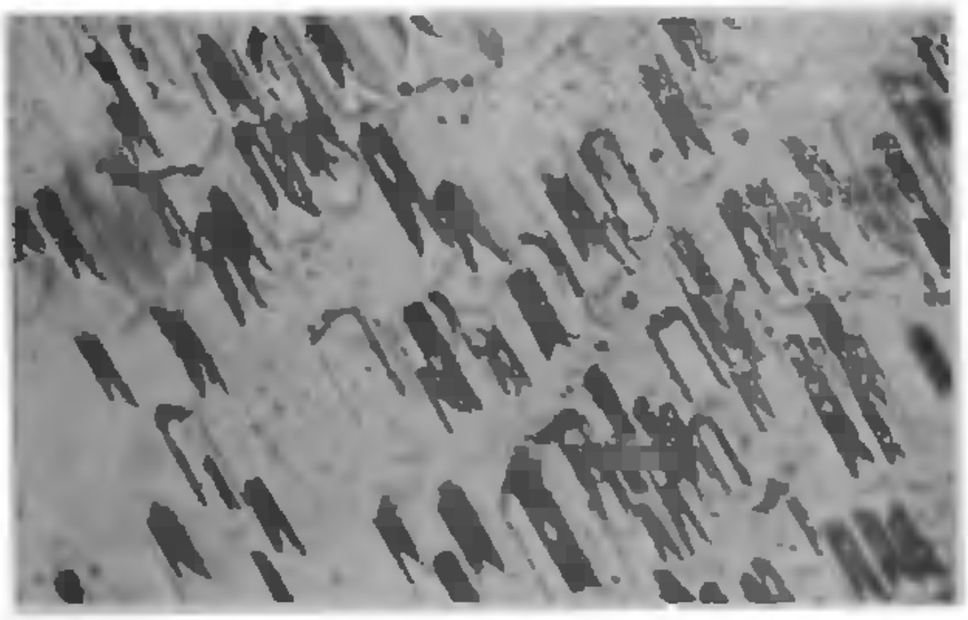

FIG. 3. Etch pits on cleavage plane of pure TGS crystal (× 300).

Copper was doped in TGS solution in the range 20 ppm to 2000 ppm. Seeds were prepared by

evaporating the solution at room temperature. Well developed disc type seeds of mass about 50 mg (0·6 cm in dia. and 0·1 cm thick) were used to grow single crystals of doped TGS. Perfectly transparent and well defined crystal (Fig. 4) of mass about 7·3 g and dimensions (3·8 × 2·4 × 0·6 cm) has been grown in 18 days. Crystals were cleaved with a sharp blade along the cleavage plane. Figs. 5 (*a*) and 5 (*b*) show one to one correspondence of the features on matched cleavage faces. Very few lines are visible in the photomicrograph. Cleavage plane is also considerably smooth. The average dislocation density in the crystal is of the order of $\sim 10^3$ per cm^2. Fig. 6 shows the dislocation etch pattern on the cleavage plane of doped TGS crystal. The results of the comparative study of pure TGS and doped TGS crystals are given in Table I.

FIG. 4. Single crystal of TGS doped with copper.

FIG. 5 *a*, *b*. Oppositely matched cleavage faces of TGS crystal doped with copper (× 350).

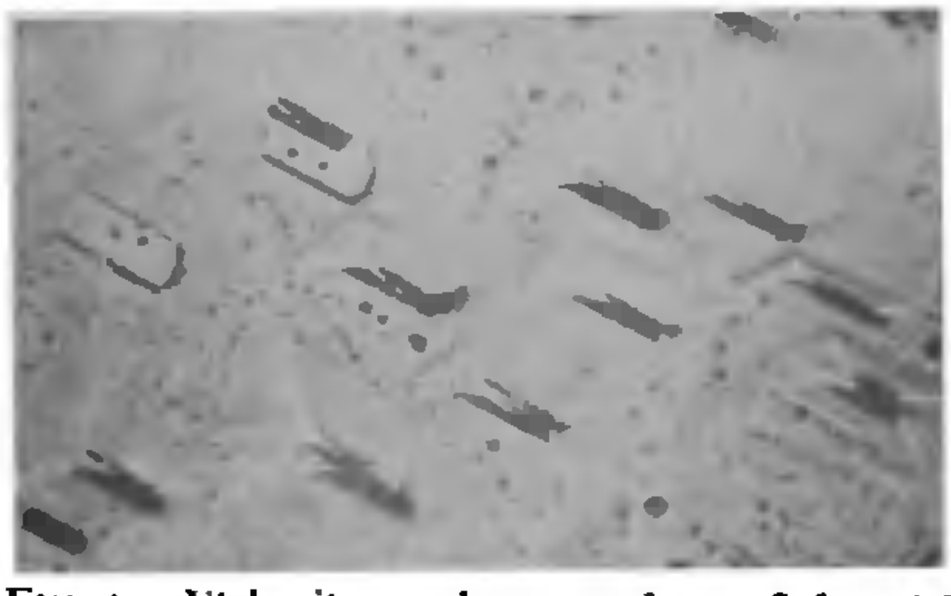

FIG. 6. Etch pits on cleavage plane of doped TGS crystal (× 300).

TABLE I

	Pure TGS	TGS doped with copper
1.	Seeds were obtained at 27° C.	Better seeds were obtained at 32° C
2.	Most of the seeds obtained were rod type and a few were plate type	Most of the seeds obtained were disc type and a few were rod type
3.	Rod type seeds gave better crystals	Disc type seeds gave better crystals
4.	Crystals were well defined but transparency was low	Cyrstals were transparent and well defined
5.	Most prominent habit faces are (100), (010), (110), (011) and ($\bar{1}$11)	Most prominent habit faces are (100), (010), (110), (011) and ($\bar{1}$11).
6.	Cleavage plane is rough	Cleavage plane is smooth
7.	Average dislocation density is $\sim 10^4$ per cm^2	Average dislocation density is $\sim 10^3$ per cm^2.
8.	Macrocracks were observed on habit faces	No cracks were observed on habit faces

It may be concluded that on doping TGS with copper, perfectness of the crystal increases. B. J. Lillicrap and J. D. C. Wood[6] have reported that on doping TGS with L-alanine, crystals are more perfect.

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