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GROWTH AND CHARACTERIZATION OF $LaMnO_3$ CRYSTALS

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ABSTRACT

The crystals of $LaMnO_3$ have been grown by single diffusion method in gel growth. The grown crystals have been analyzed by different characterization techniques. X-ray diffraction studies shows that it belongs to Perovskite crystal system having the lattice dimensions a= 5.582Å, b= 5.515Å and c= 7.763Å. The mechanical properties of grown crystals have been studied by using Vickers Microhardness tester. The surface defects were examined by adopting etching technique.

Key words: Crystal growth, Gel growth, Powder XRD, Ferroelectric crystals.

INTRODUCTION

Crystals lie at the heart of much of today's high technology. Therefore, major interest has been in the production of crystalline materials, which fulfill the device specifications. The quality of the material needed to meet these specifications can involve the production of pure single crystal variously doped. For that, a great variety of growth techniques have been developed for suitable different material. Single crystals have assumed enormous importance for both; academic, research and technology, particularly in the field of electronics. At the same time, the range of fields involved is great from electro-optics to metal corrosion, from semiconductors to magnetic bubble materials and even crystalline materials substitutes for energy conversion[1]. Hence, single crystal technology of new material with interesting properties like semiconducting ferroelectric, photoelectric, photo conducting etc., have undergone tremendous development for the past three to four decades mainly as a result of the responses to application of requirements in solid-state technology as well as in all other branches of science[2].

Crystal growth from gel is a powerful purification process and the purity of the

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crystal obtained is higher than the corresponding starting reactives. This is an advantage with respect to high temperature growth methods, where much more care should be taken in the impurity contents of the starting materials.

Crystal growth in gel technique has gained considerable importance due to simplicity and effectiveness in growing single crystals. In the present study, La_2O_3 is combined with manganese dioxide MnO_2 to form the crystals. In the present communication, we have reported the growth and characterization of $LaMnO_3$. The grown single crystals have been analyzed by different characterization techniques. The results are given here.

EXPERIMENTAL

Before growing crystals, sodium silicate gel is found preferable among the gels. To prepare gel solutions of different densities, different quantities of the stock solution (such as 10, 15, 20, 25, 30 and 35 mL) are mixed with triple distilled water such that the quantities of the final solutions are made equal to 100 mL. The densities of these solutions are determined accurately. This is taken in a black plastic container with airtight stopcock. The mixture in the container is stirred continuously to obtain the gel with water. Then the solution is filtered and if necessary, it is dehydrated and stored in a black plastic container with airtight lids. Analar grade La_2O_3 and MnO_2 of purity 99.99% and Analar grade citric acid of density 1.98 g/cc are used as the chemicals in growing the $LaMnO_3$ in gel. Good single crystals with adequate size of these $LaMnO_3$ are obtained only with the single diffusion method. The grown crystals are shown in Fig. 1.

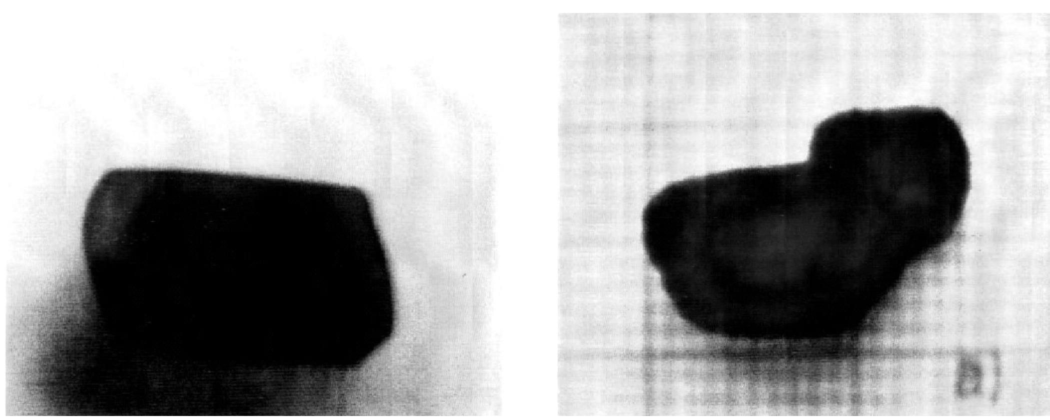

Fig. 1: $LaMnO_3$ Crystals

Characterization studies

The grown single crystals have been analyzed by different characterization techniques. The lattice dimensions of grown crystals were confirmed by X-ray powdered diffractogram using RITCHIE SIEFERT diffractometer model. The mechanical behavior of grown crystals have been studied by using a LEITZ WEITZLER micro hardness tester fitted with a Vickers diamond pyramid identer. Etching study was carried out on the (100) and (010) planes of the $LaMnO_3$ crystals using ethanol-methanol mixture as an etchant.

RESULTS AND DISCUSSION

X-ray diffraction analysis

The crystal system was identified from the powder X-ray diffraction analyses and found that it belongs to Perovskite. The calculated lattice parameters are given in Table 1. The recorded powder XRD spectrum is given in Fig. 2.

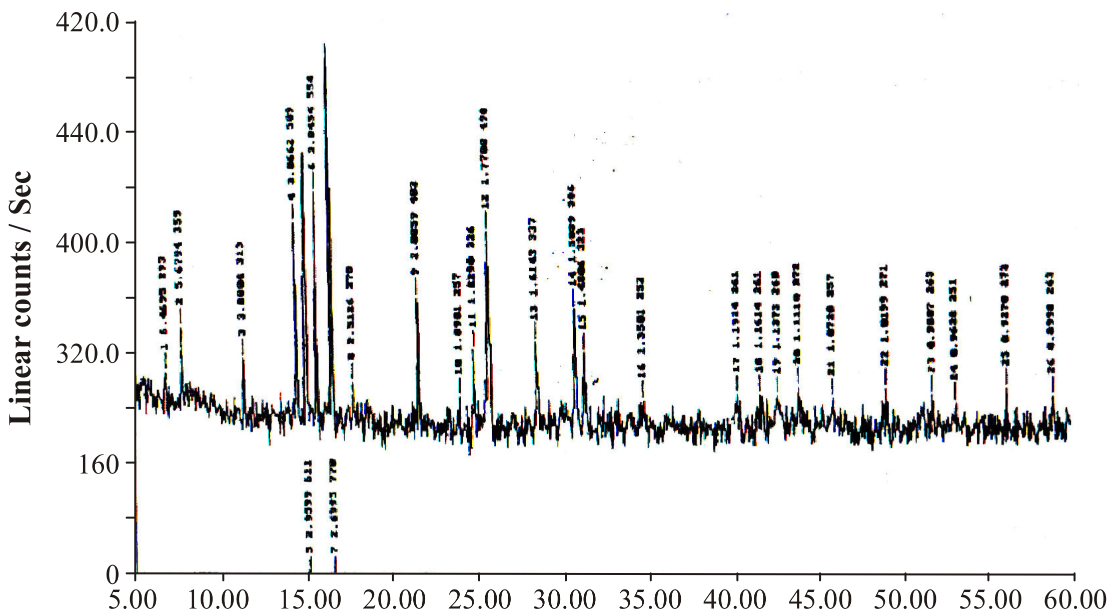

Fig. 2: XRD of $LaMnO_3$ crystals

Mechanical properties

Hardness of the material is the resistance, it offers to indentation by a much harder body. Hardness test is a method of subjecting crystal surface to relatively high pressure within a localized area. The method of measuring hardness depends on the elastic and plastic deformation characteristics of the crystal and the results obtained depend on the factors like yield point, elastic limit, elastic modulus, brittleness etc. Microhardness measurements was carried out using LEITZ WEITZLER hardness tester fitted with a diamond identor. The mechanical behavior of $LaMnO_3$ crystal were analyzed by using Vickers microhardness test at various temperatures. Selected smooth surfaces of $LaMnO_3$ crystal were chosen for this analysis. The crystal was mounted properly on the base of the microscope. Now the selected faces were idented gently by loads varying from 0 to 30 g for 10 s. The plot of Vickers microhardness vs. load is shown in Fig. 3. The hardness was calculated using the relation $H_v = 1.8544(P/d^2$, where P is the applied load in kg) and d is the diagonal length of the indentation impression in micrometer. However, with increasing load, the H_v value also simultaneously increased. The measurements performed beyond a load of 25 g resulted in severe cracks. This might be due to the release of internal stress generated locally by indentation.

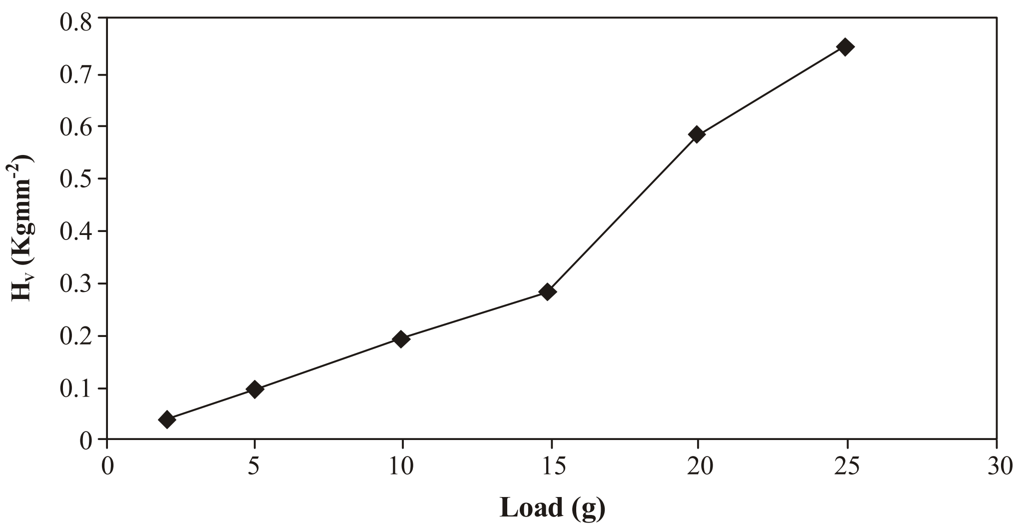

Fig. 3: Variation of Vicker's microhardness number with load

Etching studies

The crystals with defects may destroy the mechanical and electrical properties, which affect the usefulness of the crystals. Etching is one of the selective tools to identify the defects in as grown crystals. When the crystal is dissolved in a solvent, the reversal of the growth taking place by giving the well defined etches pits. The etching time depends on the solubility of the crystalline material in different solvents. Chemical etching is a simple and very powerful tool to analyze the defects present in the growing crystal surfaces.

Dislocations easily appear in crystals, especially in the initial stages of their growth[3]. In the present work, ethanol-methanol mixture is used as an etchant and etching experiment have been carried out on (100) and (010) surfaces of $LaMnO_3$. The (100) plane was dipped in the ethanol-methanol mixture for about 30-60 s and (010) plane for about 30 s and taken out and the surface etchant was dried quickly with filter paper. The recorded photographs are shown in Fig. 4(a) and 4(b). During etching, the weak surface layers have been removed. It may due to the vacancy of the atoms in some areas of the surface of the specimen. It may be due to the insufficient molecules to build the basic blocks on the surface side of the specimen.

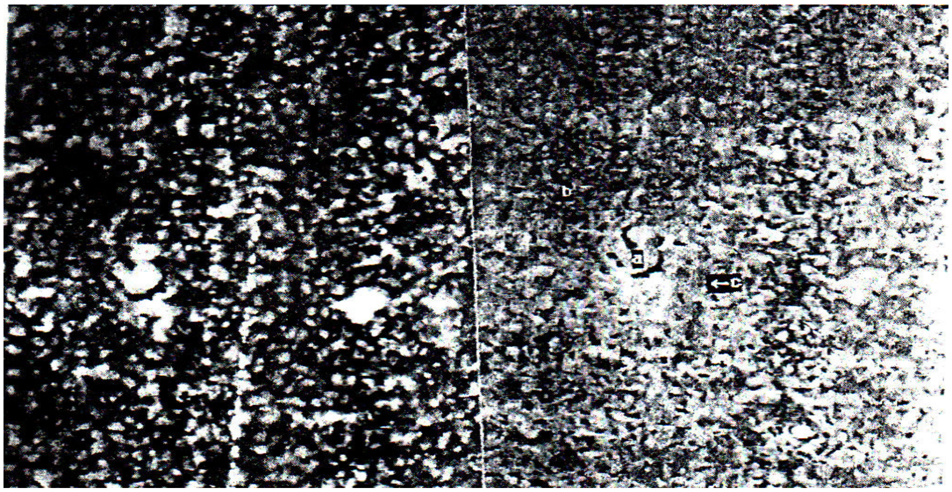

Fig. 4(a) $LaMnO_3$ etched with ethanol and methnol

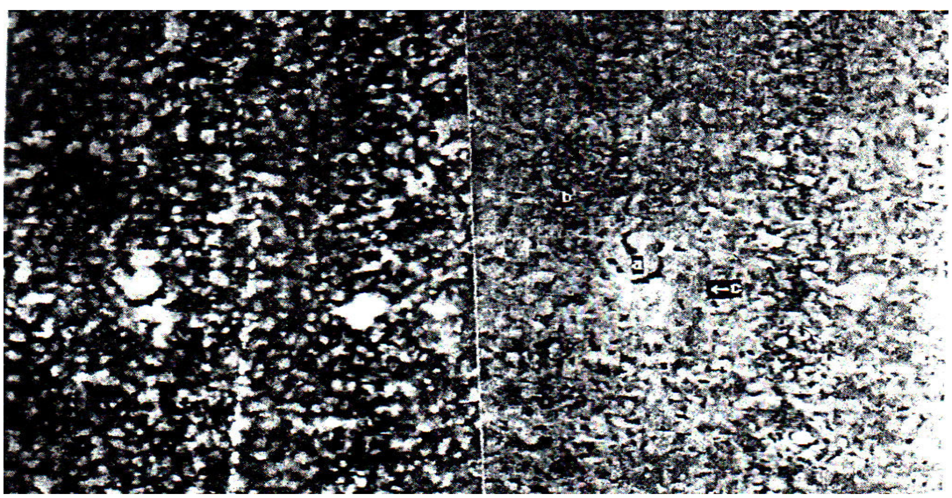

Fig. 4(b) LaMnO3 etch pattern at (010) and (100) plane

CONCLUSION

$LaMnO_3$ single crystal has been grown by single diffusion method in gel growth. The crystal system and lattice dimensions were identified from the single crystal XRD analysis. The mechanical behavior have been analyzed. Etching studies on the grown (100) and (010) planes of the crystal have revealed well defined etch pits. It may due to vacancy of atoms in the surface of the specimen.

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