Indian Journal of Pure & Applied Physics
Vol. 57, April 2019, pp. 278-284

Knoop micro-hardness studies on (110) surface of gel grown strontium tartrate di-hydrate crystal

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Received 1 May 2017; accepted 17 December 2018

The wedge shape or elongated pyramid Knoop indenter is preferred for micro-hardness anisotropy study. In the Knoop indenter, the width, volume or projected area of the indentation mark can be used as a measure of hardness by aligning the indenter along a particular direction of a surface. In the present study, the (110) surface of gel grown strontium tartrate di-hydrate crystal has been used for the Knoop micro-hardness study. The anisotropy of micro-hardness has been studied by changing the orientation of the Knoop indenter from 0º to 200º at a fixed load, which indicates one maximum and one minimum value of the Knoop micro-hardness. The variation of Knoop micro-hardness with applied load in the range of 5 g to 100 g load indicates three maxima of the Knoop micro-hardness values.

Keywords: Indentation hardness, Knoop micro-hardness, Anisotropy in hardness, Gel growth

1 Introduction

Hardness of a crystal is an important mechanical property of considerable interest. Hardness is considered as a measure of the ease to deform the crystalline solids plastically, which depends on the mobility of dislocations, their multiplication, and their interactions[1]. Moreover, it is related to the bond strength on one hand and to the defect structure on the other. It is accepted that the hardness is a measure of the resistance offered by a crystal to the motion of dislocation[2-4]. The four different indentation hardness tests are well known, viz., spherical Brinell hardness, trigonal pyramid Berkovich hardness, square pyramid Vickers hardness as well as elongated pyramid Knoop hardness[5,6]. The micro-indentation hardness is usually carried out for applied loads within 100 g. To test thin layers, surfaces with varying hardness and anisotropy in hardness the Knoop hardness is preferred. The variation of the Knoop micro-hardness with applied load is reported for phenanthrene single crystals[7], anthracene single crystals[8], calcium oxalate crystals and urinary calculi[9], single crystals and polycrystalline blanks of caesium halides[10] and $Bi_{1-x}Sb_x$ single crystals[11]. The Knoop micro-indentation hardness is also used to study the anisotropy of hardness in several single crystals, for example, phenanthrene[7], anthracene[8], lithium niobate and lithium tantalite[12], rutile[13] and epidote[14].

Strontium tartrate crystals possess several interesting properties and find certain applications, for instance, non-linear optical (NLO) property[15], dielectric, ferroelectric and piezoelectric properties[16], combustible property used in tracer composition[17], treating males suffering from diseases and conditions affecting the metabolism and structural integrity of bone[18]. The growth of single crystals of strontium tartrate in silica gel has been reported in the literature[19-21]. The mechanical property like hardness is very important for considering device application of any solid material. Having limited but important applications of strontium tartrate, its single crystals were grown by the gel technique and (110) as grown surface was selected for the Knoop micro-hardness studies. The variation of Knoop micro-hardness with orientations its long diagonal as well as the variation of Knoop micro-hardness with applied load was studied on strontium tartrate di-hydrate single crystals.

2 Experimental Technique

The glass test tubes of 2.5 cm diameter and 15 cm length were selected for the crystal growth. The gel was prepared by adding 1 molar tartaric acid solution in to sodium meta-silicate solution having specific gravity of 1.07 g/cm^3 in such a manner that the pH of the mixture could be set at 3.8. The mixture was transferred in to different test tubes to allow the gel to set. The gel was set in two days. 1 M, 25 mL

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*Corresponding author (E-mail: hojethva@rediffmail.com) strontium chloride solution was poured gently on the set gel without disturbing its surface. Good quality transparent single crystals of the maximum length 6 mm were obtained. Figure 1 shows the growth of crystals in a test tube.

Thermo-gravimetric analysis (TGA) was carried out at Regional Sophisticated Instrumentation Centre, Nagpur using Perkin-Elmer (USA) TGS-2 set up from room temperature to 950 ºC at heating rate of ±2 to ±100 ºC/min in inert atmosphere. From TGA, it was found that two water molecules were associated with the crystals. The grown crystals were analyzed by powder XRD using PW1710 based Diffractrometer set up at Regional Sophisticated Instrumentation Centre, Nagpur with Fe Kα radiation. The unit cell parameters were calculated by using programme REFEDT. BAS. Software and found to orthorhombic with unit cell parameters as: a = 9.48 Å, b = 10.96 Å and c = 9.46 Å. Figure 2 shows habit of grown crystal with assigned planes.

The crystallographic planes were assigned by using the reported habit of grown crystal reported in the literature[22,23]. Crystal with smooth as grown (110) face were selected after careful examination through optical microscope attached with Knoop indenter and then the Knoop indentation tests were carried out. The etching study on the smooth as grown (110) face crystal by 0.1 M NH_4Cl exhibited crystallographically aligned etch pits and from the traces of the slip lines the primary slip direction was identified. The etching study results were in correspondence to the earlier reported work in the literature[24].

Fig. 1 — Growth of crystals in test tube.

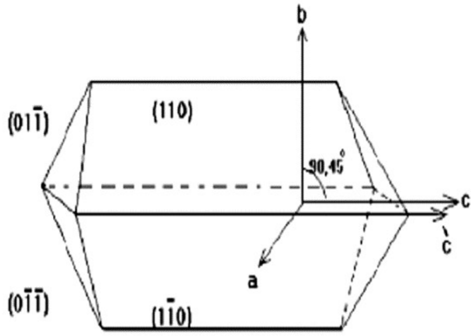

Fig. 2 — Habit of grown crystal.

The selected (001) face of crystal was indented by the Knoop indenter attached to a CZ-vertical microscope available at Applied Physics Department, M.S. University of Baroda, India as well as the Leitz-indenter with automatic read out of impression marks and the microprocessor based automatic calculation of the Knoop hardness data using the facility available at Department of Applied Chemistry, University of Strathclyde, Glasgow, U. K. The long diagonal was used in calculating the Knoop hardness number using the formula: $KHN = 14228.8 \times (P/d^2)$ kg/mm^2**,** where, P is the applied load in gram and d is the mean long diagonal length in micrometer. The indentation time of 10 s was kept constant. Minimum five indentations were made for each load as well as each orientation value. The standard deviation values are calculated using well known formulations and shown as bars with average value data points in the plots drawn. Figure 3 shows the typical photomicrograph of the Knoop indentation on the (110) surface of strontium tartrate di-hydrate single crystal at 5 g load.

For anisotropy in hardness study, the Knoop indentations were made at different orientations of the long diagonal of the Knoop indenter on the selected (110) surface of the crystal. The orientations were changed from 0º to 200º in the steps of 10º at constant

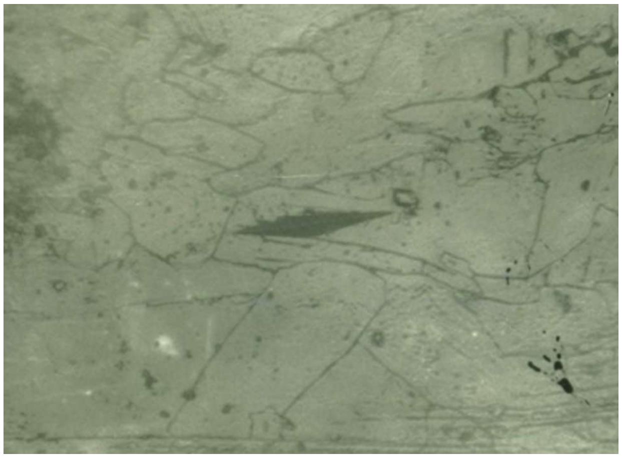

Fig. 3 — Knoop indention on (110) surface.

load of 15 g. At least five indentations were made at each orientation on the same selected face of grown crystal. The average values were obtained and standard deviation was calculated and shown as bars in the plot Knoop hardness versus orientation of the indenter.

To study the variation of Knoop micro-hardness with load, the indents were made at room temperature with loads varying from 5 g to 100 g in the steps of 5 g. At least five indents were made at each load on the same crystalline face of the selected crystal and average value was obtained along with the standard deviation.

3 Results and Discussion

In the gel growth technique, one reactant is impregnated in the gel and the other reactant is the supernatant solution poured on the gel. The controlled diffusion of the reactants occurs through the porous gel medium and reaction between the reactants occurs resulting in to nucleation and growth of the crystals[20,25,26]. The effect of various parameters such as gel density, gel pH, concentration of solutions, etc has been reported by several workers[27,28]. These growth parameters control the pore size of the gel in majority and hence the passage of the nutrients for the growth of crystals. In the present study, the values of the gel density and the gel pH were selected by performing several growth runs with different values of growth parameters and the optimum values were obtained for the growth of good quality crystals.

Basically, the Knoop indenter is suggested for the studies of hardness anisotropies. An important feature of the Knoop hardness test is that the hardness value is dependent on the orientation of the major axis of the indenter in a given plane as well as on the orientation of the plane with reference to the principle axis of anisotropy[29]. In the case of Knoop indenter, the width, volume or projected area of the indentation can be used as a centro-symmetric measure of hardness with indenter aligned along a particular direction on the surface. Sergeant and Page[30] considered anisotropy as the ratio of maximum to minimum hardness values, which is greater for materials with a limited number of slip systems. For any material, this effect is prominent if a wedge-shape Knoop indenter is used rather than pyramidal indenter. It has been suggested that in the plane of the surface and for a given orientation θ of the indenter, the magnitude of the elastic recovery strain is given by the ratio of the lateral compressive stress Y induced by the loaded indenter and the effective Young modulus E in the plane of the test surface. Both of these quantities vary with orientation on the surface. The indentation contracts by an amount proportional to Y_θ/E_θ in each direction. For Knoop indentations, however, very little compressive stress is expected in the line of the long diagonal and, therefore, little elastic recovery is found in the diagonal irrespective the orientation of the indenter on the surface. Whereas, for Vickers indentations, the maximum compressive stress is expected normal to the faces and there is always a strong component of lateral compressive stress in line with each of the diagonals and hence some elastically driven recovery can be observed along widths and diagonals both.

The factors controlling the Knoop indentation hardness in isotropic and anisotropic materials are considered[30]. In perfectly isotropic materials, a number of effects are controlling both the load-on and load-off dimensions and shapes of hardness indentations, for example, the yield stress, work hardening, elastic recovery and creep. In this case, only the shape of the indenter which determines the different responses around indentation, for example, differing strains resulting in different amount of elastic recovery along the long and short diagonals of the Knoop indentation; but this does not generate change in indentation shape as the indenter is rotated and hence no hardness anisotropy. However, in anisotropic materials, these controlling factors may well display individual anisotropies, including the orientation of crystallographically discrete slip systems. The differential surface pile-up of material displaced from indentation can confuse the estimation of true slip anisotropies, whereas both pile-up and differential elastic recovery can confuse the estimation of yield stress anisotropies. Therefore, it has been suggested by Sargent and Page[30] to define these effects as intrinsic and extrinsic effects. The intrinsic effects, which are the various responses of the material itself to the indentation being made, can be characterized as deformation character and geometry. The extrinsic aspects, which subsequently change the size and shape of the impression finally measured, are characterized as elastic recovery, surface topography of the displaced material and errors concerned with the measurements. Since, the pile-up or sink-in itself is resulted from the slip geometry; it is arguable whether or not this effect should be classified as intrinsic.

The mutual relationship between the operative slip system and the measured hardness anisotropy indicates that the explanation is lying in the behaviour of dislocations and the bulk plastic flow. Therefore, the same type of anisotropy behaviour is observed for the crystals have slip system common. In the model proposed by Daniels and Dunn[31], they considered the hardness as an inverse function of the ease of slip initiation. They assumed that, for each indenter facet, the effective force responsible for deformation is a tensile force parallel to the steepest slope of the facet. In this analysis the deformation is considered as small cylinder pulled in tension parallel to this effective force. From this model an effective resolved shear stress (ERSS), which is directly proportional to the product of the cosines of three angles, viz., (1) between the slip direction and the effective force, (2) between the normal-to-the-slip plane and the effective force, and (3) between the direction in the slip plane perpendicular to the slip direction and the direction in the indenter-facet plane perpendicular to the effective force. For any slip system and indenter orientation these angles can be determined and an effective resolved shear stress calculated. However, the Daniels and Dunn model was not successful in all analysis and certain discrepancies were observed. The modifications in the stress-state assumptions of Daniels and Dunn model were made by Feng and Elbaum[32] and Garfinkle and Garlick[33]. According to their suggestions instead of considering a tensile force parallel to the indenter facet as the effective deformation force, this force is, instead, a compressive force which is perpendicular to the indenter facet. Earlier, several workers like Daniels and Dunn[31], Garfinkle and Garlick[33] and Partridge and Roberts[34] showed clearly that the anisotropy in hardness could be determined by the crystal structure and the primary slip systems. Brookes *et al.*[35] showed that for fluorspar crystals with slip system {001} $\langle 1\bar{1}0\rangle$ on (001) planes revealed the minimum value of hardness along the $[1\bar{1}0]$ directions and also for the rock salt crystals with the {110}$\langle 1\bar{1}0\rangle$ slip system on {110} plane exhibited the low value of hardness along $[1\bar{1}0]$ directions. Sasaki and Iwata[36] studied the anisotropy of micro-hardness on (001) plane of anthracene single crystals using the Knoop indenter. The number of hardness maxima for complete rotation is constant with the crystallographic symmetry of the plane indented[5,31]. In agreement of this, Miller[37] found one maximum of the Knoop micro-hardness for the crystal orientation on the (110) habit face of explosive material PETN (Pentaerythritol tetranitrate) crystals. The hard direction was found to be parallel to $[1\bar{1}0]$ and the soft direction was at ± 15º from [001] direction. On the other hand, the anisotropy in micro-hardness on (111) cleavage plane of bismuth single crystals was reported by Joshi[38] using Vickers micro-indentation hardness. It was concluded that the slip direction $[10\bar{1}]$ was the softest one and the symmetry of the plane gave three maxima within the full orientation of 360º.

In the present investigation, the indentations were made at different orientations of the long diagonal of the Knoop indenter on the (110) face of strontium tartrate di-hydrate crystal. The orientations of the long diagonal of the Knoop indenter were varied from 0º to 200º in the steps of 10º at constant load of 15 g. The variation of Knoop micro-hardness at different orientation of its long diagonal on (110) as grown surface has been shown in the plot of Fig. 4 with the error bars or standard deviations.

In this plot, one maximum value and one minimum value of the Knoop hardness is observed within 0º to 180º of orientation, which suggests two fold symmetry of the (110) face. Generally, the slip systems in the orthorhombic system are {201}[101], {401}[101], {110}<110>, {001}[010], {010}[001] and {101}<101> or {101}[010], {110}[001] and {201}[001][39]. In the crystalline solids, within the range of stress in engineering application, the component of the stress normal to the slip plane does not influence the slip. The slip cannot take place

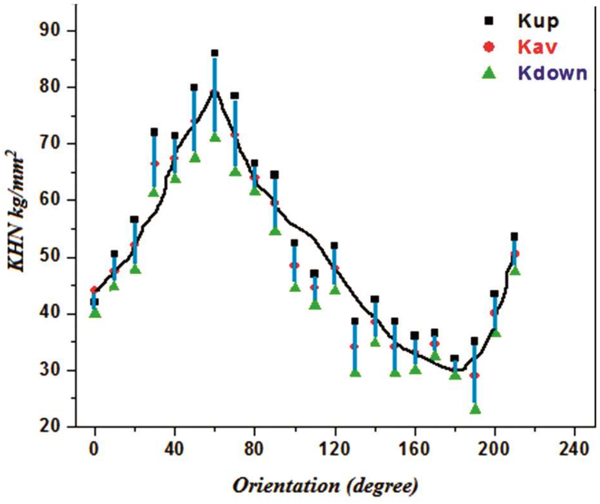

Fig. 4 — Variation of knoop hardness with orientation.

under normal stress on a plane; the stress needs shear (tangential) component on a plane to affect the slip process. Therefore, the slip process is considered in terms of the shear stress resolved on the slip plane in the slip direction. The hardness is inversely proportional to the ERSS, i.e., the direction with lowest ERSS is the hardest one. Moreover, the geometry of the slip system, the crystallographic plane and the direction of slip combine to produce a modification in a resolved shear stress. This is discussed in detail elsewhere[40, 41]. Brookes *et al.*[35] calculated the effective resolved shear stress (ERSS) for the (001) face of fluorspar structure having $\{001\}\langle 1\bar{1}0\rangle$ slip system. It was found that the greatest ERSS value was observed in the $[1\bar{1}0]$ direction making it the softest direction, the lowest value was in the [100] direction making it the hardest direction. The similar results were reported for (100) plane Epidote mineral crystals having slip system (100)<001> by Din *et al.*[14]. In general, the slip direction is the most closely packed direction of the lattice. Moreover, the anisotropy in the Knoop hardness with crystal orientation and the ERSS profile has been investigated by Joshi[42] and Gallaghar *et al.*[43] in calcite crystals. As the slip results from the dislocation motion, those dislocations require the lowest energy will move readily. A higher shear stress is required to produce slip on planes having lower packing density. The strontium tartrate crystal crystallizes in orthorhombic symmetry[44] and the {110}<110> slip system is operative. The [110] direction is softest direction with lowest value of the Knoop hardness. These results correspond to the work reported on PETN by Miller[37].

The hardness values at low loads exhibit quite different nature and many times disputable also as discussed by Shah and Shah[45] considering various factors affecting the variation. In some reports[46,47] an increase in the hardness value is obtained due to the methods of preparing specimen since the surface layers are hardened by polishing process[48], but in the other report decrease in hardness due to vibrations at low loads[49]; whereas some claim no variation in hardness[50]. Usually, the low loads are taken as standard loads of 1, 2, 5, 10, 50 and 100 g, but there is a large gap between the 10, 50 and 100 g loads and explored well earlier by several authors and reported the variation in hardness with low loads exhibiting several humps[7,45,51], which has been explained by them on the basis of work hardening and yield processes incorporating dislocations in different slip systems. It is generally accepted that the hardness relates to the elastic moduli and slip systems of crystals[52]. The variation in the Vickers micro-hardness with load has been studied widely on large number of materials[20,52,53]. Shah[51] studied the Vickers micro-indentation hardness on adamantine crystals and found that the maximum Vickers micro-hardness of 3 kg/mm^2 occurred for a load of 16 g, then the micro-hardness dropped at 20 g load and then it increased and reached the plateau region over the load of 35 g. The author[51] suggested that the slip took place of (111) [101] dislocation up to the load of 16.9 g interacting and strengthening material and then the sudden dip in the curve was due to the creation of new dislocations which then interacted to yield the subsequent rise in the curve for loads 20 g to 30 g. The constant hardness value over 35 g was due to the work hardening of the crystals. Later on, Marwaha and Shah[54] obtained two peaks in the micro-hardness versus load plots on the (001) cleavage plane of biphenyl single crystals and with the help of crystal structure and the molecular positions within they attributed the first peak to the slip in [010] direction and the second peak due to the slip in [101] direction. Vaidya and Shah[7] observed three peaks in the plot of variation of the Knoop hardness with load for phenanthrene single crystals. They attributed the peaks to the dislocations of types $(20\bar{1})$ [010] and (100)[010] and conjectured that the first two nearby peaks were due to splitting of the $(20\bar{1})$ [010] dislocation in to two partials of (201) [010] and $(20\bar{1})$ [010]. Recently, Jani *et al.*[11] reported variation of the Knoop hardness with load in $Bi_{0.95}Sb_{0.05}$ crystals and found that the Knoop hardness increased initially then exhibited a dip and finally gained the plateau region.

The Vickers micro-hardness study of strontium tartrate crystals was reported by Patel and Arora[24] and found that the hardness increased with load and became constant nearly at 90 g load. Moreover, the Vickers micro-hardness studies were reported on manganese doped strontium tartrate tetra-hydrate crystals[55] and barium mixed strontium tartrate[56], where the Vickers micro-hardness increased with applied load. In most of the micro-indentation hardness studies standard loads at large intervals are taken, for example, 25 g, 50 g and 100 g, but in the present study the loads are chosen in combinations such that the load at smaller intervals can be applied. However, the entirely different nature of variation in

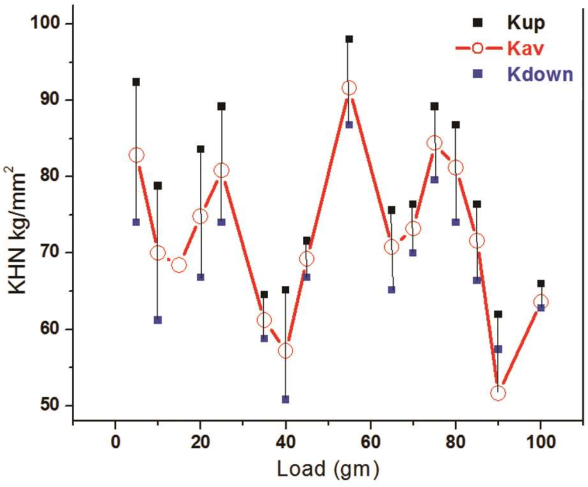

Fig. 5 — Variation of Knoop micro-hardness with load.

the Knoop micro- hardness with load is obtained in the present study. Figure 5 shows the variation of the Knoop micro-hardness with load on (110) face of strontium tartrate di-hydrte crystal.

The plot exhibits three peaks of the Knoop micro-hardness at values of 82 kg/mm^2, 92 kg/mm^2 and 84 kg/mm^2 at applied loads of 25 g, 55 g and 75 g, respectively. By considering orthorhombic structure of strontium tartrate crystals, it is conjectured that the three peaks in the Knoop hardness versus load curve are due to the dislocations of (110)[110], (110)[001] and (101)[101] and their slipping gives three different peak positions. This needs further validation by alignment of etch pits around the indentation mark and observation of slip traces on both as grown and deliberately deformed crystal to get idea of active slip planes and further augmentation by the X-ray topography. However, no attempt is made at this juncture due to experimental limitations.

4 Conclusions

The variation of the Knoop micro-hardness with orientation of long diagonal of the indenter in different directions on (110) face of strontium tartrate di-hydrate crystal suggests one maximum value and one minimum value of micro-hardness within 0º to 200º orientation, which indicates two fold symmetry of the (110) habit face with [110] slip direction as the softest direction. The variation of Knoop micro-hardness with load from 5 g to 100 g, in the steps of 5 g, produces three peaks of micro-hardness values, which are conjectured due to the slip of (110) [110],(110)[001]and (101)[101] dislocations.

Acknowledgement

The authors are highly thankful to Prof J N Sherwood (Formerly at University of Strathclyde, Glasgow, UK), Prof B S Shah (Retired, HOD, Physics Department, Saurashtra University, Rajkot), Dr D R Joshi (Department of Applied Physics, M S University of Baroda, Vadodara) for useful discussion and providing necessary facilities and Prof H H Joshi (HOD, Physics Department, Saurashtra University, Rajkot) for his keen interest. The authors are thankful to UGC for SAP and DST for FIST.

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