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Crystal Growth of Napthalene ($C_{10}H_8$) crystals using bridgemann-stockbarger technique

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ABSTRACT

A vertical Bridgemann Stockbarger crystal growth workstation was designed and fabricated indigenously. It consisted of a single zone z-axis oriented resistively heated furnace capable of reaching 400˚C. A ceramic insulation blanket is arranged around the concentric vicinity in order to insulate to perfection. Naphthalene is an organic compound with formula $C_{10}H_8$*. As an aromatic hydrocarbon, naphthalene's structure consists of a fused pair of benzene rings. Naphthalenes substituted with combinations of strongly electron-donating functional groups, such as alcohols and amines, and strongly electron-withdrawing groups, especially sulfonic acids, are intermediates in the preparation of many synthetic dyes. They are also used as dispersants in synthetic and natural rubbers, and as tanning agents (syntans) in leather industries, agricultural formulations (dispersants for pesticides), dyes and as a dispersant in lead–acid battery plates. Napthalene also has good luminescence, fluorescence and dielectric properties. Crystals of Napthalene were grown by Bridgmann-Stockbarger technique and were characterized for their structural, optical and spectroscopic properties.*

Keywords:Vertical Bridgemann, crystal growth, napthalene crystal

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INTRODUCTION

The Bridgman–Stockbarger technique is named after Harvard physicist Percy Williams Bridgman and MIT physicist Donald C. Stockbarger. Bridgmann initiated the idea of a two zone arrangement which will initiate crystal formation and obviously the growth of the crystal, wheras the Stockbarger technique involves the translation of the ampoule with the charge horizontally or vertically. These two methods are integrated in order to be primarily used for growing single crystal ingots (boules), and which can be used for solidifying polycrystalline ingots as well. The method involves heating polycrystalline material above its melting point and slowly cooling it from one end of its container, where a seed crystal is located. A single crystal of the same crystallographic orientation as the seed material is grown on the seed and is progressively formed along the length of the container. The process can be carried out either in a horizontal or vertical geometry.[1-3]

The Bridgman method is a more popular way of producing certain crystals from volatile and rapidly vaporizing materials for which the Czochralski process is more difficult. The difference between the Bridgman technique and Stockbarger technique is subtle: while a temperature gradient is already in place for the Bridgman technique, the Stockbarger technique specifies the pulling the boat through a temperature gradient to grow the desired single crystal. When seed crystals are not employed as described above, polycrystalline ingots can be produced from a feedstock consisting of rods, chunks, or any irregularly shaped pieces once they are melted and allowed to re-solidify. The resultant microstructures of the ingots so obtained are characteristic of directionally solidified metals and alloys with their aligned grains. The Bridgman furnace works with three temperature zones. The upper zone is arranged with temperatures slightly above the melting point of the material to be grown. The lower zone is arranged to have a temperature profile fairly below the melting point of the substance, and an insulated adiabatic zone in between, acts as a baffle between the zones. The Bridgman technique is a method of growing single crystal ingots or boules. It is a popular method of producing certain semiconductor crystals, such as gallium arsenide, II-V Crystals (ZnSe, CdS, CdTe) and BGO, where the Czochralski process is more difficult.

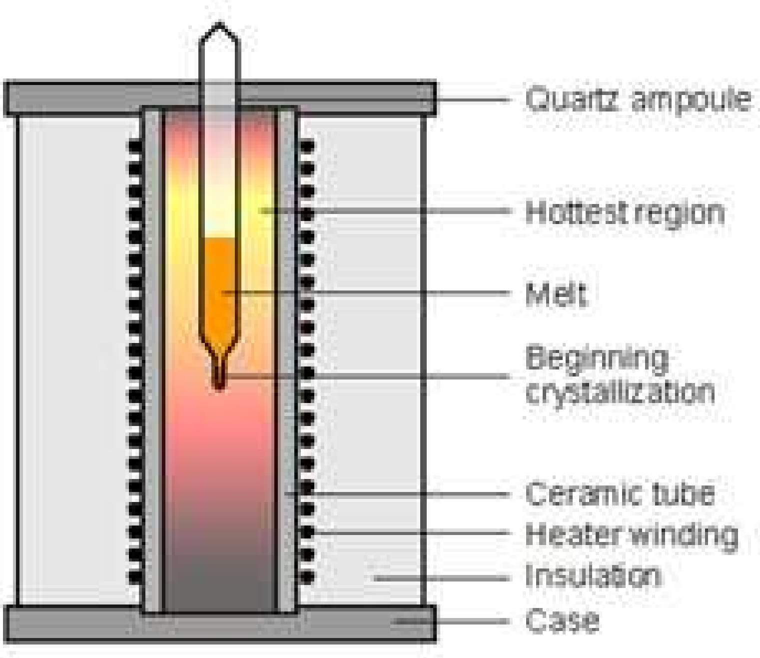

Fig. 1: Bridgmann-Stockbarger schematic set-up

CRYSTAL GROWTH OF NAPTHALENE

Approximately 200 gm of naphthalene ($C_{10} H_8$) of CDH brand, laboratory grade is filled inside an open Borosil ampoule. The top surface of the ampoule is tightly sealed and hooked on with a wire connection to the pulley and DC motor which offers upward translation in the z-axis by slow rotation. The DC motor with a good torque is vertically arranged with the Bridgmann Stockbarger workstation and it is capable of pulling the ampoule slowly inside the furnace without touching the bottom part, sealed shell or any of the sides of the furnace [4]. When the ampoule is arranged in the centre of furnace, the power supply is switched on thereby activating the temperature increase. The initial temperature of the furnace is set at 30°C (room temperature) and the temperature is gradually increased in steps 1°C per minute. When the furnace temperature reaches the melting point of naphthalene, the material inside the ampoule reaches the molten state. Further the vaporization of the naphthalene which is now in complete homogenized melt form is avoided by immediately activating the cooling cycle using the thermostat. Now crystallization of naphthalene starts from molten state (liquidus) to solid state (solidus). Now the temperature of the furnace was gradually decreased in the steps of 1°C per second. The ampoule is pulled upward by using the pulley of the DC motor from the furnace arrangement and finally after complete lifting of the ampoule the crystal of naphthalene is achieved and harvested [5-9].

Fig. 2: Crystal Growth of Napthalene

ABSORPTION AND TRANSMISSION STUDIES OF NAPTHALENE

Absorption spectroscopy is a powerful spectroscopic technique that measures the absorption of radiation, as a function of frequency or wavelength, due to its interaction with a particular sample. The sample absorbs energy, i.e., photons, from the radiating field. The intensity of the absorption varies as a function of frequency, and this variation is the absorption spectrum.

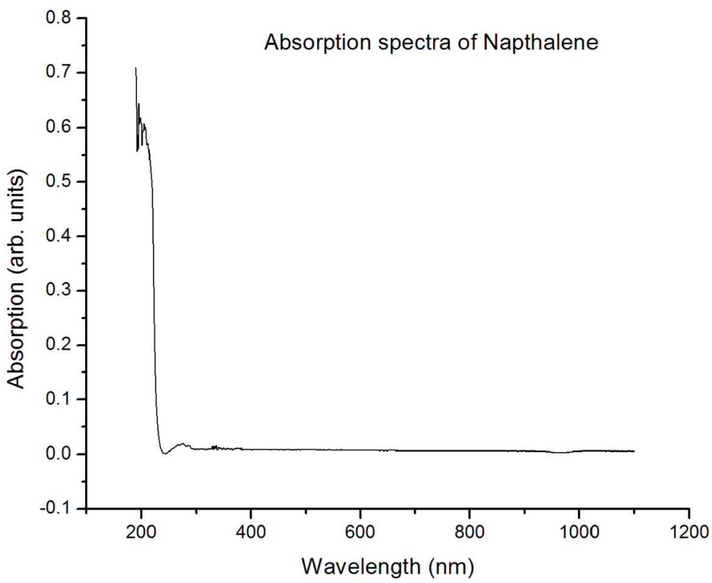

Fig. 3: Absorption spectrum of Napthalene

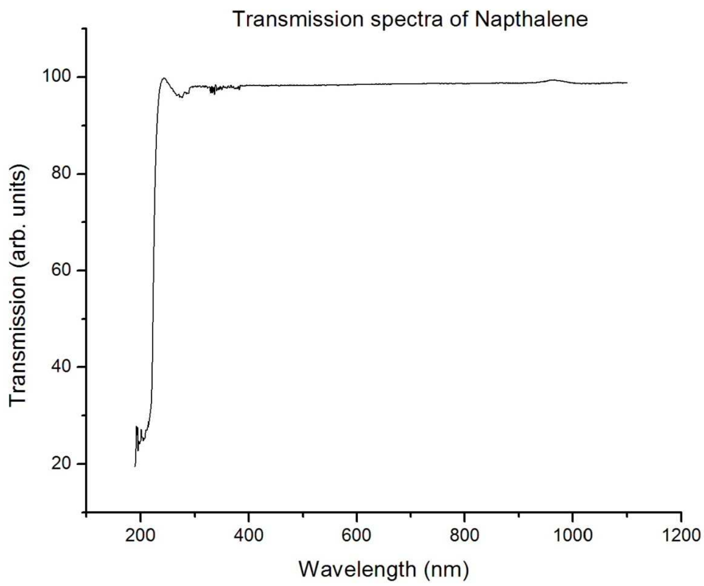

Fig. 4: Transmission spectrum of Napthalene

Absorption spectroscopy is employed as an analytical chemistry tool to determine the presence of a particular substance in a sample and, in many cases, to quantify the amount of the substance present. There are a wide range of experimental approaches to measuring absorption spectra. The most common arrangement is to direct a generated beam of radiation at a sample and detect the intensity of the radiation that passes through it. The transmitted energy can be used to calculate the absorption. The source, sample arrangement and detection technique vary significantly depending on the frequency range and the purpose of the experiment.

Absorption spectra for Napthalene was engaged on a Perkin-Elmer Lambda spectrophotometer. It was found to have a total absorption characteristic till 250nm in the ultra-violet region. From 250 to 1200nm there was total transmission which attributed to the good crystalline quality of the sample.

Absorption and transmission spectra represent equivalent information and one can be calculated from the other through an inverse or reciprocal transformation. A transmission spectrum will have its maximum intensities at wavelengths where the absorption spectrum is weakest because more light is transmitted through the sample. An absorption spectrum will have its maximum intensities at wavelengths where the absorption is strongest. The transmission spectra was taken in the Perkin Elmer spectrophotometer from 200nm to 1200nm. Complete absorption was observed from 200nm to 300nm. From 300nm to 1200nm there was total transmission of around 95% which is a qualitative evidence of the quality of the crystal.

FTIR INVESTIGATIONS OF NAPTHALENE

The FTIR spectral analysis is shown in the Fig. 3. There is a sharp and less intense peak at 3046 cm^{-1} and is assigned to the C–H stretching of naphthalene. Absence of peaks in other energy region above 2000 cm^{-1} supports the purity of the naphthalene crystals. The ring skeletal vibration of naphthalene is assigned from the well-resolved peaks between 800 and 1600 cm^{-1}. The high intense sharp peak at 776 cm^{-1} is assigned to CH bending. In the higher energy region, the CH stretching appears as an intense sharp signal at 3198 cm^{-1}. In the finger print region below 1675 cm^{-1}, there are few peaks which are seen to have the partners in the FT-IR spectrum. Since naphthalene has center of symmetry, the mutual exclusion principle can be applied for accounting the presence and absence of peaks in the finger print region. In the present measurements, the vibrations corresponding to the peaks 1579, 1494, 1125 and 600 cm^{-1} are observed in the FT-IR spectrum, illustrating these vibrations are IR active.[10-11]

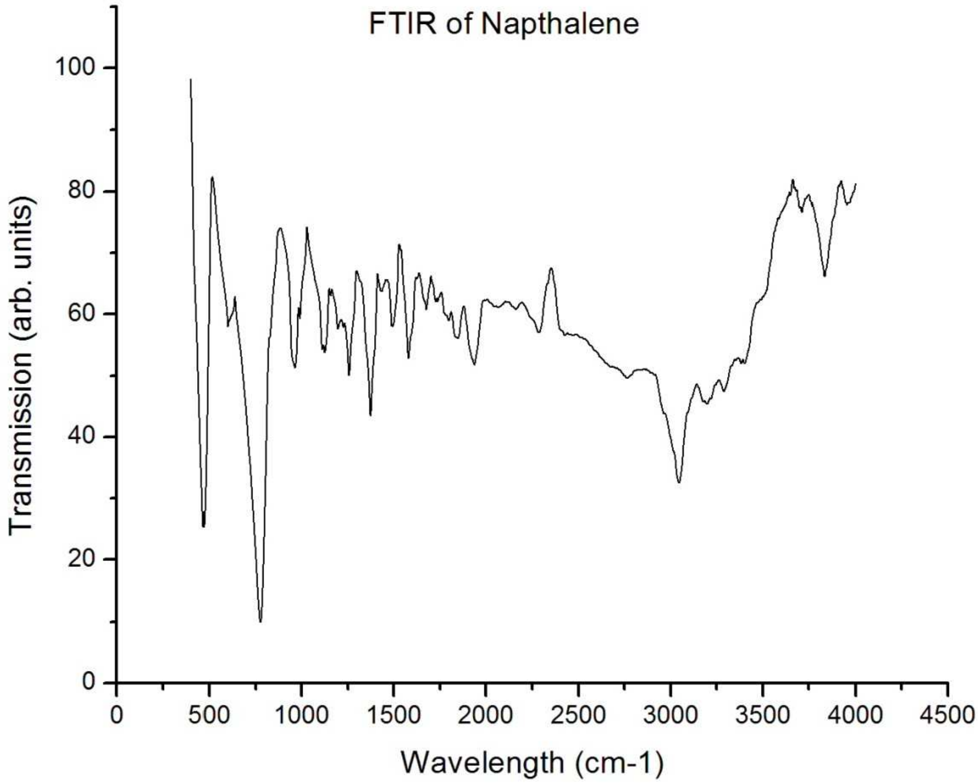

Fig. 5: FTIR spectrum of Napthalene

RESULTS AND DISCUSSION

The Bridgmann-Stockbarger crystal growth workstation was fabricated and Napthalene crystal was successfully grown and characterized. The Absorption and Transmission studies confirmed qualitatively the nature of the crystal grown rather highlighting on the quality of growth. The FTIR measurements gave a thorough explanation of the bonds and essential alignments of the atoms in each crystals.

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REFERENCES

[1] K. Barraclough (Eds.), *Advanced Crystal Growth*, Prentice-Hall, Englewood Cliffs, NJ, **1986**.
[2] N. Karl, in: H.C. Freyhardt (Ed.), *Crystals: Growth Properties, Applications*, Vol. 2, Springer, Berlin, **1980**.
[3] J.D. Wright, *Molecular Crystals*, Cambridge University Press, UK, Second Edition, **1995**.
[4] W.R. Wilcox, *J. Crystal Growth*, **1970,** 7**:** 203-207

[5] R.C. Sangster, J.W. Irvine, *J. Chem. Phys.*, **1956,** 24**:** 670-675
[6] J. Bleay, R.M. Hooper, R.S. Narang, J.N. Sherwood, *J. Crystal Growth*, **1978,** 43**:** 589-594.
[7] A. Uedono, M. Tachibana, M. Shimizu, M. Satoh, K.Kojima, *Jpn. J. Appl. Phys.*, **1996** 35: 3623-3632
[8] D.W.J. Cruikshank, *Acta Cryst*., **1957,** 10: 504-511.
[9] François Cardarelli, *Materials Handbook: A Concise Desktop Reference,* Springer, Amsterdam, **2008.**
[10] Wells, A. F., *Structural Inorganic Chemistry (5th Ed.),* Oxford: Clarendon Press, **1984**.
[11] S. Sivananthan, N. Neelakanda Pillai, N N Pillai and C. K. Mahadevan, *Archives of Physics Research*, 2012, 3 (3):239-244