International e-Conference on Recent Trends in Nano-Materials and Its Applications-2021
In Association with International Journal of Scientific Research in Science and Technology
Volume 9 | Issue 2 | Print ISSN: 2395-6011 | Online ISSN: 2395-602X (www.ijsrst.com)

Studies to Determine the Young's Modulus of Monolithic Silica Aerogels

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ABSTRACT

Aerogel materials are not only superinsulating but also a sound proof. Such materials are very fragile in nature besides it we have examined the elastic properties of methyltrimethoxysilane (MTMS) based silica aerogels. This current research work focuses the elastic behaviour of the monolithic silica aerogels. The technique of interferometry was applied to study elastic constant of aerogels. It was observed that due to mechanical stressing, number of fringes localised on the aerogel samples and these fringes were used to determine the Young's modulus (Y) of such materials. Here, we report a new mode for determination of 'Y' of aerogel monoliths.

Keywords: Mechanical Stressing, Young's Modulus.

I. INTRODUCTION

Silica aerogels are nano-structured materials with the bulk density as low as 0.02 g/cm^3 and high porosity (> 98%) [1]. Due to the high porosity and very less solid content, determination of the various mechanical properties of the silica aerogels is the major challenge. Though methods like three point flexural techniques, Vickers and knops tests have been applied for the measurement of the mechanical properties of the aerogels, but application of even very small loads (~0.25 N) results in the cracking of the aerogel samples [2,3]. Therefore efforts have been made in the past to use non-destructive technique as like sound velocity measurements for this purpose [4, 5]. There are no reports available on the use of holographic interferometry for the determination of mechanical properties of the aerogels.

Holographic interferometry has been widely accepted as a viable tool for non-destructive testing of materials. It permits the qualitative & quantitative study of minute changes in the object contours [6]. We report here the use of double exposure holographic interferometry (DEHI) [7] to study the surface deformation of mechanically stressed aerogels. DEHI has proved to be an advantageous than the other holographic techniques in the study of transient phenomenon [8]. In this technique, there is comparison of a stressed surface state relative to its unstressed state causes interference fringes to be observed on the object, which gives information of the object deformation with a very high precision [9,10]. As this technique is enough sensitive to the determination of the order of wavelength of source [He-Ne LASER 6328 Å], application of a small stress can give rise to interferometric fringes and hence the sample under test remains intact, reusable and crack free.

II. EXPERIMENTAL

2.1. Preparation of Monolithic Silica Aerogels:

Two stage sol-gel processes was used. Alcogels were prepared by hydrolysis and polycondesation of tetraethoxysilane (TEOS). Initially, TEOS was dissolved in methanol (MeOH) in order to make it soluble in 0.05 NH_4F, Methyltrimethoxysilane (MTMS) was added to this mixture to make the aerogels hydrophobic as well as diffusely reflective. The mixture was stirred for 10 minutes and the resulting homogenous sol was transferred to 25 ml pyrex glass test tubes. The test tubes were made air tight to inhibit the evaporation of MeOH from the sol. The gelation took place at an ambient temperature of 27 ºC. In order to prevent shrinkage and cracks in the alcogels, excess amount of MeOH was poured in the test tubes after gelation. All the gels were aged at 25 ºC for 24 hours. The aged alcogels were dried supercritically in an autoclave. The details of autoclave drying conditions were given in this publication [11].

To synthesize aerogels with different physical characteristics, molar ratios of TEOS:MTMS:H_2O:NH_4F was kept constant at 1:1:4:3.6 X 10^{-3} respectively and MeOH/TEOS molar ratio (M) was varied analytically from 12 to 16, as it was observed in an earlier study that variation of M values affects the bulk density of aerogel [12-13].

2.2. Holographic Experimental Set-up:

The off axes double exposure holographic interferometry technique was used to record the holograms. In this technique, continuous comparison of the surface displacement relative to its initial position causes interference pattern observed on the object. Surface, which gives information about the object [5,6].

All the optical components used in the recording were arranged on the vibration isolation system [7]. The special type of the vibration isolation table is prepared in holography laboratory from local equipments in low cost. This system avoids all the vibrations reaching on the top of the table from ground. The cylindrical samples of aerogels having ˜10 mm radius and ˜30 mm height were used to study the surface deformation using DEHI technique. The aerogle sample was illuminated by 5mW He-Ne laser [λ = 6328 Å]. High quality beam splitter [70:30] was used to split the laser beam.

We used the mechanical stressing technique to study deformation. To study the effect of mechanical stressing, the object (aerogel) was placed on a rigid black painted wooden block and a weight (e.g. 1 g) was placed on it. The special pulley arrangement was used to unload the samples. The hologram of the object was recorded in this stressed state on the holographic plate. Then without disturbing the whole system, the load was removed from the object and the same holographic plate was exposed to this natural unstressed state of the object. The sufficient exposure time was adjusted depending upon the reflection from the object in both cases.

III. DETERMINATION OF YOUNG'S MODULUS

There are three different methods for the determination of Young's modulus of different solid objects. The Y of a long, thin wire can be calculated by the following equation,

$$Y = \frac{MgL}{\pi r^2 l} \tag{1}$$

Where, M = Mass applied,
L = Length of wire,
r = radius of wire,
l = elongation produced.

In this work, we have calculated the elongation 'l', by holographic method by calculating the deformation of the silica aerogel sample after application of the load. Hence by using the holographic method the equation (1) can be modified as follows,

$$Y = \frac{MgL}{\pi r^2 a} \tag{2}$$

Where, ‘a’ = surface displacement of the sample. The surface displacement of sample can be calculated by the following geometry.

3.1) Geometry for in- plane displacement of a rigid object:

To view the object from a distance R, in the following figure,

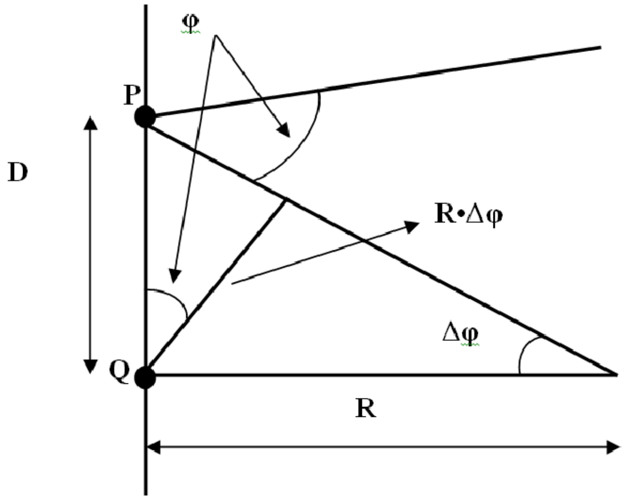

Figure 1: Geometry for in- plane displacement of a rigid object

Where, R = distance of object, φ = viewing angle.

The separation of fringes on the object is given by,

$$D = \frac{R\Delta\varphi}{\cos\varphi} = \frac{R\lambda}{a\cos(\varphi)\sin(\varphi - \partial)} \qquad (3)$$

For the silica aerogel samples, the movement after the application of load is parallel to surface, hence in above equation (3), δ = π/2. Hence equation (3) becomes,

$$D = \frac{R\lambda}{a\cos^2\varphi} \qquad (4)$$

Where, ‘a’ = surface displacement of the sample

$$\therefore\ a = \frac{R\lambda}{D\cos^2\varphi} \qquad (5)$$

Here, D = Fringe separation, λ = wavelength of the source, φ = viewing angle, R = Distance of object. Using equation (2) the values of the ‘Y’ of the silica aerogel samples can be calculated.

IV. RESULTS

4.1. Fourier Transform Infrared Spectroscopy (FTIR):

FTIR study of the silica aerogel was carried out.

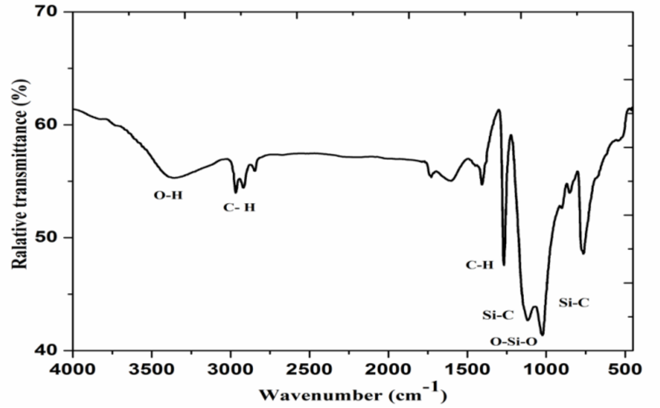

Figure 2: FTIR Study of the silica aerogel

Several characteristic absorption bands were observed in the range of 450 cm^{-1} to 4000 cm^{-1} indicating the presence of methyl groups [14]. The broad absorption band observed at 1080 cm^{-1} is a characteristic of the Si-O-Si bond present in samples. The absorption bands were observed at 2950 and 1400 cm^{-1} due to stretching and bending modes of C–H bond and the deeps observed at 765 & 1265 cm^{-1} due to the Si–C bonds. The 1265 cm^{-1} peak indicates the presence of the Si–C bonding [15]. The absorption deeps at 1600 and 3400 cm^{-1} corresponding to the polar –OH bonds

4.2. Determination of Young’s modulus of silica Aerogel:

We have determined the ‘Y’ of the silica aerogel samples having different bulk densities and thermal conductivities of the silica aerogel monoliths. These values are reported in the table no. 1. We have used the silica aerogels having the five different densities. First the values of the surface deformations were calculated. Values of surface deformations of all the samples were also reported in table no. 1

Sample	Density 'ρ' (g/cm^3)	Thermal conductivity 'k' (W/m K)	Applied load in 'g'	No of fringes.	Deformation (x 10^{-3} cm)	Youngs modulus 'Y' (x10^5 dyne/cm^2)
M 9	0.1808	0.0023	2	7	5.9531	2.51
			5	13	6.6468	7.74
			10	15	7.7518	14.49
			15	17	20.23	7.68
M 17	0.1973	0.0029	1	4	3.100	2.41415
			2	12	9.300	1.61076
			5	21	10.89	4.7287
			7	23	11.37	6.1470
			10	40	14.32	9.1529
M 25	0.1566	0.0019	0.5	9	19.801	2.9946
			2	42	47.619	4.9404
M 26	0.1408	0.0019	1	7	15.90	1.531
			2	9	18.73	1.666
			3	16	46.50	1.569
M 31	0.1346	0.0016	0.5	7	18.049	1.10494
			1	10	37.35	2.8416
			2	27	75.73	2.8910

Table No:1. Physical and elastic properties of monolithic silica aerogels.

All the results have shown that as load on the sample increases, surface deformation also increases. All the values of the 'Y' calculated are reported in table no.1 and all the values are in good agreement with the standard values. The photographs of some of the silica aerogel samples are shown here.

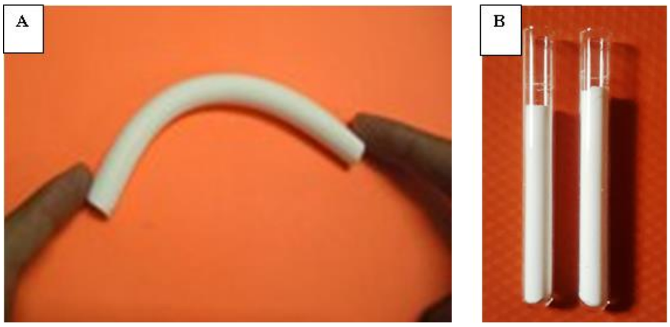

Figure 2: Photographs of synthesized silica aerogel monoliths,
[A] Flexible silica aerogel showing bending behaviour.
[B] Silica aerogels in 25 ml test tubes.

V. CONCLUSION

We have applied successfully DEHI technique for determination of 'Y' of sol-gel derived porous silica aerogel monoliths. These monoliths exhibited very low thermal conductivity. Above mentioned properties were achieved by quantifying the sol-gel chemistry and parameters during the supercritical drying of the silica aerogels. Usually, the derived porous materials are rigid in nature but this work demonstrated the new measure in the synthesis of porous, elastic and insulating silica aerogel monoliths.

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