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To cite this version:

D. Samatowicz. INTERNAL FRICTION OF SOLID SAMPLES COVERED WITH SURFACE LAYER OF LIQUID CRYSTAL. Journal de Physique Colloques, 1987, 48 (C8), pp.C8-525-C8-529. <10.1051/jphyscol:1987882>. <jpa-00227186>

HAL Id: jpa-00227186
https://hal.archives-ouvertes.fr/jpa-00227186
Submitted on 1 Jan 1987

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INTERNAL FRICTION OF SOLID SAMPLES COVERED WITH SURFACE LAYER OF LIQUID CRYSTAL

D. SAMATOWICZ

Gdańsk Technical University, Department of Technical Physics and Applied Mathematics, PL-80-952 Gdańsk, Poland

Abstract - Internal friction measurements were made for glass and metal samples covered with thin layer of liquid crystal MBBA, NPOOB and CN. The measurements were performed with the torsion pendulum in the frequency range 1 ± 40 Hz and heating rate <10 °C/min. The thin liquid crystal layer on the surface of solid sample causes the appearance of distinct maxima in the internal friction spectrum $Q^{-1}(T)$ and large changes of modulus $G$. The temperatures of melting and phase transitions for the investigated liquid crystal are close to the temperatures of the maxima in the spectrum $Q^{-1}(T)$.

Introduction

In recent years some experimental results determining the effect of surface layer on internal friction $Q^{-1}$ and elasticity modulus $G$ of metals [1,2,3,4] have been presented mainly by Polish authors at European and Polish conferences. The final conclusion from the previous investigations of internal friction carried out on samples of metal covered with a thin layer of hydrocarbon, oil or Wood's alloy is the connection between the internal friction maximum appearing on curve $Q^{-1}(T)$ and the melting temperature of the layer material.

In all the published papers the problem of the interpretation of the remaining part of spectrum $Q^{-1}(T)$ at higher temperatures is still unsolved. Since there are no theoretical papers concerning the adsorption of organic compounds and structure of thin layers of such substances the problem seems to be very difficult to explain even in the experimental way.

In this work internal friction measurements were made for samples covered with thin layer of known liquid crystals—MBBA, NPOOB and CN.

Experimental

The measurements of the internal friction were carried out on a simple torsional pendulum of K type in frequency range 1 ± 40 Hz. The measurement principle consisted in the determination of logarithmic decrement $\delta$ of free vibrations of the sample $Q^{-1} = \delta/\pi$ and the frequency connected with elasticity modulus for shearing $f^2 \sim G/\chi$. Both quantities were continually registered from nitrogen temperature to high temperatures /below the desorption temperature of the covering substance/ with heating rate <0.5 °C/min. The vibrations with deformation amplitude of order $10^{-5}$ were analyzed. The samples used here were rod-shaped of 20 ± 50 mm length.
and diameter 0.5 ≈ 2 mm made of technological copper, platinum, steel and silicate glass. The samples, after the initial measurement of the background were covered with a liquid layer firmly adsorbed on the sample. The layers of liquid crystal were obtained by lubricating. The layer thickness was calculated from the weights of the sample without and with liquid crystal. The layer thickness was of order of a several micrometers. The covering layers were typical liquid crystals - MBBA, NPOOB and CN.

The Investigation Results and Discussion

A thin liquid layer set on the surface of a metal or glass causes the appearance of distinct maxima in the internal friction spectrum $Q^{-1}(T)$. With the maxima correspond large changes of elasticity modulus $G$. The temperatures of the appearance of maxima in spectrum $Q^{-1}(T)$ are close to the temperatures of the phase transitions of liquid crystals.

First, the highest maximum frequently appears at the lowest temperatures and is connected with the melting temperature of liquid crystal independently of the phase to which the liquid crystal transforms.

The next maxima appear at temperatures close to the temperatures of the corresponding phase transitions. Fig. 1 shows the $Q^{-1}(T)$ and $f^2(T)$ curves for Pt sample, clean and covered with NPOOB. For pure platinum, in the investigated range of temperatures there are no maxima - only a small temperature increase of the background is observed.

For the sample with NPOOB, in addition to the high maximum corresponding to the liquid crystal melting, two small maxima appear at temperatures where the transitions $S \leftrightarrow N$ and $N \leftrightarrow I$ occur. The similar situation can be observed for Cu samples covered with MBBA - Fig. 2 and 3. Here, besides the two maxima at the temperatures of about 295 K and 315 K /i.e., the melting temperature of MBBA and phase transition $N \leftrightarrow I$/ additional two slight maxima appear at about 220 K. At this temperature the liquid crystal is the solid phase. The calorimetric measurements [5] stated the metastable phases occurring at this temperature, and their properties are closely connected with heat treatment of the sample /cooling rate and range/. On Fig. 4 the $Q^{-1}(T)$ and $f^2(T)$ curves for Cu sample covered with monotropic unpolar liquid crystal CN are presented. In addition to the two maxima corresponding to the normal phase transformations K 353,5 Ch 365 I, the maximum below the melting of this liquid crystal appears during heating the sample. The temperature of the appearance of maximum /~350 K/ is close to the Ch,S transition temperature which is observed during cooling.

CN is the liquid crystal very slowly crystallizing from the smectic phase. Probably, the internal friction maximum appearing below the melting point during heating may be connected with the slight supercooled part of the substance in the smectic phase, now that the maximum is higher when the sample was cooled shortly after setting CN from isotropic phase on the Cu surface /before the proper measurement/.

The result presented here corresponds to one-day cooling at room temperature. For the samples which were cooled only for 3-4 hours, the height of this maximum is nearly the same as for the maximum appearing at the melting point.

Table 1 shows the temperatures of phase transitions for the liquid crystals presented here and the temperatures of the corresponding maxima of internal friction.
Table 1

<table>
<thead>
<tr>
<th>Liquid crystal</th>
<th>Temperatures of phase transitions</th>
<th>Temperatures of internal friction maxima</th>
</tr>
</thead>
<tbody>
<tr>
<td>MBBA</td>
<td>Crystal/nematic phase</td>
<td>295 (\pm) 300 K</td>
</tr>
<tr>
<td>K 295 N 320 I</td>
<td>nematic phase/isotropic phase</td>
<td>295 K</td>
</tr>
<tr>
<td>/p-methoxybenzylidene p-butylaniline</td>
<td>320 K</td>
<td>315 K</td>
</tr>
<tr>
<td>NPOOB</td>
<td>Crystal/smectic phase</td>
<td>325 K</td>
</tr>
<tr>
<td>K 322 SA 334 N</td>
<td>smectic/nematic phase</td>
<td>325 K</td>
</tr>
<tr>
<td>341 I</td>
<td>nematic/isotropic phase</td>
<td>336 K</td>
</tr>
<tr>
<td>/4-n-pentylphenyl-4'-n-heptyloxythiolbenzoate/</td>
<td>341 K</td>
<td>340 K</td>
</tr>
<tr>
<td>CN</td>
<td>crystal/cholesteric phase</td>
<td>352 K</td>
</tr>
<tr>
<td>K 350.5 353.5 Ch 365 I</td>
<td>cholesteric/isotropic phase</td>
<td>365 K</td>
</tr>
<tr>
<td>/pelargonate cholesterol/</td>
<td></td>
<td>360 /heating/</td>
</tr>
<tr>
<td>W5</td>
<td>stable/nematic phase</td>
<td>340 /cooling/</td>
</tr>
<tr>
<td>nematic crystal</td>
<td>nematic/isotropic phase</td>
<td>340 /cooling/</td>
</tr>
<tr>
<td>K 253 N 335 I</td>
<td></td>
<td>335 K</td>
</tr>
</tbody>
</table>

From the experimental results it follows that the maxima obtained at the frequency 1 \(\leq\) 5 Hz are the highest, of course when the same dynamics of measurement was applied.

Only the slight lowering of the temperatures of maxima is observed at the N\(\rightarrow\)I transitions for nematics, but the shape of the spectrum /a number of maxima/ is not connected with the kind of substrate. It is confirmed by the \(Q^{-1}(T)\) curves presented on Fig. 5 determined for different samples covered with the W5 composition which exhibits the nematic phase in the wide temperature range from about 260 K to 335 K. The previous measurements performed with the W5 composition indicated that the height of the first maximum at the temperature about 220 K is strictly proportional to the amount of the liquid crystal put on a sample[6].

All the observed internal friction maxima are accompanied by the large changes of \(f^2(T)\). The greatest changes can be found when the layer material transforms from solid to liquid phase. The \(f^2(T)\) changes connected with the other maxima seem to be related to the temperature variation of viscosity of a layer material.
The primary results obtained for the liquid crystals allow to connect the observed phenomena with the phase transformations occurring in the liquid crystal layer. These processes are not typical of the relaxation process studied by the internal friction method but some of the phase transformation theories applied to the description of phase transformations in solids may bring a considerable contribution to the present investigations. It should be pointed out that the internal friction method is very sensitive and that it is possible to apply this method for determining the properties of the layer material. An attempt of the mathematical description of processes responsible for the observed maxima will be presented in the next paper.

**Fig. 1.** $Q^{-1}(T)$ and $f^2(T)$ spectra for clean Pt /----/ and Pt sample covered with NPOOB /--/ at $f_{293K} = 17$ Hz.

**Fig. 2.** $Q^{-1}(T)$ and $f^2(T)$ spectra for clean Cu /--/- and Cu sample covered with MBBA /-----/ at $f_{293K} = 2.5$ Hz.

**Fig. 3.** $Q^{-1}(T)$ and $f^2(T)$ spectra for clean Cu /- -/- and Cu sample covered with MBBA /----/ at $f_{293K} = 33$ Hz.
This work is partly supported in the frame of CPBR 8.12 Problem.