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Misinterpretations of DSC Data

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Scientific Notes

Misinterpretations of DSC Data

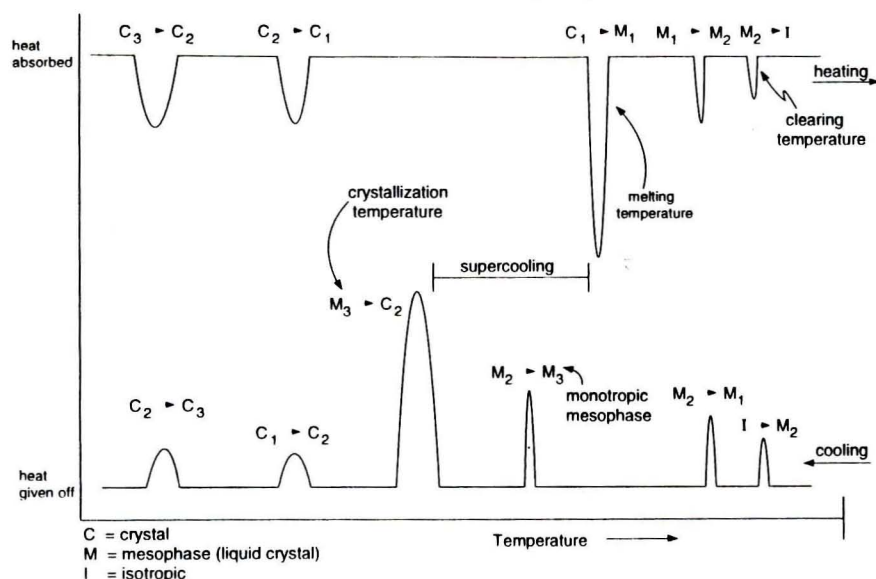
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Several recent examples in the liquid crystal literature of incorrect interpretations of DSC scans suggest that a review of the basics of interpreting DSC scans is needed. I have drawn two DSC scans for a theoretical material in the figure shown here to illustrate my points.

It is a well known fact that most melts of both inorganic and organic compounds supercool when cooled, which means that they crystallize at a lower temperature than they melt. This is what makes it possible to observe monotropic liquid crystalline phases. It is also quite common to observe crystal-to-crystal changes which often are mistaken for mesophase transitions. The possible combinations of crystal changes that can occur are quite varied; one example is shown in the figure.

Usually the melting and crystallization transitions have the largest ΔH values in a DSC scan. When they do not, it is usually because there are several crystal changes. Peaks below the largest ΔH peaks should always be suspected to be crystal forms until other data such as x-ray are available to support a mesophase identification. It is not unusual to find that immediately reheating a cooled sample gives a different DSC scan. This is because the sample has not yet returned to the same crystal form that occurred in the original scan. Often allowing the sample to set overnight will accomplish this by giving a repeat of the original scan.

A better definition for monotropic phases is that they are phases observed below the melting temperature, not just phases ob-



Sometimes different crystal forms give different melting temperatures, making it possible for a mesophase to be enantiotropic with the lower melting temperature and monotropic with the higher melting temperature. On the other hand, liquid crystalline phases rarely supercool (I have never seen one that does). Thus, if a phase that is formed on cooling is reheated and returns to the previous phase within 0.6° , it is probably a mesophase. If it does not, it is probably a crystalline phase. Sometimes supercooling to a crystalline phase is small, making it difficult to differentiate between a crystalline phase and a mesophase. Crystallization temperatures depend on how the sample is cooled and tend to vary from run to run. Fluctuations in mesophase transitions are smaller unless the sample decomposes.

served on cooling. Monotropic phases can be reheated and should return immediately to the previous phase on reheating, confirming that they are indeed mesophases and not crystals. Sometimes monotropic phases occur near the melting temperatures but always below it. Also, it is unusual to observe crystallization occurring at the same time as a mesophase forms.

The identification of mesophases by microscopic textures is an extremely useful tool but its limitations should be kept in mind. Identifying more highly ordered smectic phases is not always easy for even the highly trained person. Such a person gains the necessary skills by looking at many hundreds of compounds with a wide variety of phases. There is no

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Meeting Reports

2nd Ukrainian Liquid Crystal Conference, Christmas 92, Kiev, Ukraine

Report from A A Gerasimov, *Institute for Single Crystals, Kharkov, Ukraine.*

The liquid crystal community from 12 scientific and industrial groups of Ukraine was represented by 53 delegates who presented 23 oral reports. The scientific area of the conference programme was wide-ranging and involved various topics reflecting the interests of the research groups.

Much of the work was on surface phenomena in liquid crystals. Particularly, T Marusii (Kiev) presented reports on the orientation of liquid crystals by the UV-induced easy axis in surface film of polyvinylcinnamate. Yu Resnikov (Kiev) talked about the surface orientation effect in nematics due to light-induced changes of boundary conditions in orienting films doped with dye molecules. The theory of the influence of anchoring energy on phase behaviour and configurational transitions of nematics in cylindrical pores was presented by A Kiselov (Chernigov).

Some reports dealing with non-linear optical effects in liquid crystals were also presented. G Klimusheva (Kiev) reported results of fast-time resolution hologram recording in induced cholesterics and V Reshetnyak (Kiev) presented a paper on the molecular theory of anomalous low light-induced reorientational threshold effects in nematics doped by dyes.

A few papers were presented on the theory of liquid crystals. V Kuzmin (Odessa) talked about the quantitative aspects of chirality and the correlation between dissymmetry functions and twisting power for various types of OAD. A Gerasimov (Kharkov) presented a molecular theory of smectic mesomorphism that took into account both steric and dispersive interactions as well as the effects of the conformational dynamics of mesogenic molecules.

The results of theoretical investigations into the cholesteric-nematic transition due to an electric field for induced cholesterics was presented by Yu Terentieva (Kiev). It was shown that broken concentration uniformity leads to a power singularity for helical pitch instead of the logarithmic dependence predicted by de Gennes. S Shiyankovskii (Kiev) talked about some anomalies of conductivity in liquid crystals under irradiation. (continued on p 7)

Meeting Reports

10th School of Physics and Applications of Liquid Crystals and Single Crystals, Zakopane, Poland, 12-17 October 1992.

Conference report from the organiser, Józef Zmija, Institute of Technical Physics WAT, 00908 Warsaw, Poland.

This traditional biennial meeting of scientists working in chemistry, physics, technology and applications of liquid crystals and single crystals was organised by the Institute of Technical Physics WAT, Warsaw and supported by the Committee of Scientific Research (KBN), by the International Society for Optical Engineering (SPIE) and by the Committee of Crystallography

Polish Academy of Sciences. The Conference was held in Zakopane, the Tatra mountains Polish resort.

The programme included overview lectures and also oral and poster presentations of original results. Approximately 140 participants attended the Conference from Poland, Russia, Italy, UK, Belgium, Ukraine, Belorussia and Lithuania.

J A Janik (Kraków, Poland) delivered the main lecture about the first "liquid-crystalline" Nobel prize which went to P G de Gennes in 1991. Some studies on the synthesis and properties of new liquid-crystalline materials were reported. R Dabrowski (Warsaw, Poland) presented new materials for STN displays containing boroxanes, and poster presentations included new materials and metal-organic compounds.

Physical properties of liquid crystals were discussed in some interesting contributions. A S Sonin (Moscow, Russia) presented an overview of ordering and viscous-elastic properties of lyotropic liquid crystals. F Simoni (Napoli, Italy) reported on the optical phase shift induced by PDLC. J Jazdyn (Poznan, Poland) delivered a wide overview of the dielectric properties of liquid crystals. A Adamczyk (Warsaw, Poland) discussed possibilities for computer modelling fundamental structures and defects in liquid crystals. Diamagnetic properties and elastic constants in isothiocyanobenzenes were reported by Z Raszewski (Warsaw, Poland). An overview of x-ray investigations in liquid crystals by the freely suspended film method was given by J Przedmojski (Warsaw, Poland). E L Wood (Exeter, UK) described the electrooptical response of a thin



Photo courtesy of Andrzej Makowski, Warsaw, Poland.

liquid crystal layer in the pre-transitional regime, and G V Klimusheva (Kiev, Ukraine) reported on the properties of dynamic holography gratings in chiral liquid crystals.

The most interesting theoretical presentation, among others, was the theory of ferro-electric liquid crystals as a micropolar medium, presented by Cz Rymarz (Warsaw, Poland). Numerous contributions were devoted to LCD applications. This included the status of flat screens (Zielinski and Zmija, Warsaw Poland); PDLC properties and applications (Zmija, Klosowicz and Raszewski, Warsaw Poland); liquid crystalline optical devices (Nowinowski-Kruszelnicki, Warsaw Poland); and LCD for laptop computers (A Smirnov, Minsk Belorussia). There were also some "exotic" subjects. For example an application of cholesteric liquid crystals in allergy tests (Zuber et al, Warsaw Poland) and some molecular aspects of gamma radiation effects on cholesterol derivatives (Klosowicz and Zmija, Warsaw Poland).

A variety of problems were also discussed in the solid state part of the School. M A Herman (Warsaw, Poland) delivered a lecture about superconductor superlattices, their crystallisation and applications. A Rogalski (Warsaw, Poland) presented possibilities for introducing new ternary alloy systems for infrared detectors e.g. InAsSb and HgZnTe, better than conventional HgCdTe. The effect of organic dopants on growth kinetics and some properties of crystals grown from aqueous solutions was presented by V A Kuznetov, (Moscow, Russia). Superlattices and quantum wells for infrared optoelectronics were reported by F F Siao (Kiev,

Ukraine), and some new semiconductor materials and their properties were presented by SW Svechnikov (Kiev, Ukraine). Despite the fact that the School sessions stretched from 9.00 am to 9.00 pm, participants found time to join in many social events such as an excursion to the mountains. Foreign participants visited the former Polish capital, Kraków. Before the Conference began there were also visits to the laboratories of the Military Technical Academy, in which the physical and chemical properties of liquid crystals are investigated. The contributions presented during the School will be published in a separate SPIE issue No 1845, and selected papers will be published in MCLC. ■

Scientific Notes

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substitute for this visual experience. Without it, even photographs of known phases can look similar. Additionally, both mesophases and crystalline phases can show mosaic textures and they can look similar. The trained eye can differentiate between crystals and meso-phases to some degree on how the phases grows — a really slow or fast one means the phase is crystalline. Microscopy is extremely useful in differentiating mesophases from crystalline phases when used in conjunction with DSC but visual experience is necessary to make the correct assignments.

My goal here is to convince the reader of the need to eliminate the known possibilities before concluding that a liquid crystalline phase is present; not that exceptions to what I have said cannot occur. ■