Draft Meeting Minutes
ASC OP TF 6 – IR Materials
In joint session with
SPIE IR Materials Working Group
Thursday, August 16, 2012 , 14:00 – 16:30 PDT

Santa Rosa Room
Marriott Marquis and Marina Hotel
331 W. Harbor Drive
San Diego, CA

1. Welcome and Introductions

Gary Weise called the joint meeting to order at 14:08 with a brief introduction of all the attendees. There were over 30 attendee's (see list in attachment) representing 23 companies or labs.

2. Approval of the agenda

Gary Weise presented a revised version of the agenda and indicated the intent of this meeting was to add new participants, continue to discuss the issues, gather input from the participants and organize the plan of action. He hoped we could get to the point of assigning committee leaders for the major topics in this task.

Dave Aikens motioned to accept the agenda as modified, the motion was seconded by David Hasenauer and the motion passed.

3. Informational papers

The first speaker on the agenda was Peter Brown from Lattice Materials to speak about the process of growing and testing crystalline IR optical materials. For Ge and Si the method used to grow the crystals is the Czochralski process. The material is melted in a crucible and a seed is used to grow and draw the material from the melt. When a dopant is added to the melt there is a gradient of distribution of the dopant from top to bottom. The dopants can be either n-type or p-type depending on the requirement of the electrical properties of the materials. The dopant level starts as some concentration and enters the crystal material thru diffusion. Most of the dopant does not make it into the crystal. As the pull out of the crucible continues the dopant increases in concentration. This changes the resistivity of the material from low on top to high on bottom. The optical properties can change with the level of dopant. Usually the most transmissive material is undoped. Index measurements over temperature are complex to do. The crystal growers do not know how much the dopant effects the index of the material. Typically resistivity vs absorption is a U shaped curve, so there is a sweet spot in the desired dopant level. There is also a radial gradient in the dopant. The amount of dopant for optical materials is usually very small. Based on the dopant level the index of Si at the same temperature may vary in the 3rd decimal place. Inclusions, in the form of gas bubbles do occur as O_2 molecules can move into the melt from the crucible. The use of float zone glass eliminates these but it is a more expensive process. The interface is hidden when the bubble occurs so they are hard to detect and they are difficult to find after the crystal is formed. Transmission measurement for scatter can be made, but it requires polishing the top and bottom of the crystal. There is an instrument (they?) use to measure the IR transmission it costs about $250K to build. If only the index and transmission are specified than undoped material is the most well known. But even for undoped materials there are other impurities that are present in the initial melt material. One would have to use a mass spectrometer to identify these impurities. There are residual thermal stresses in the material that effects index and birefringence but the amount of this is still unknown. Repeatability from ingot to ingot is very good in resistivity but unknown in the actual dopant level.

The second scheduled informational speaker was not present, so his talk was skipped.

4. Working group organization and meetings

The meeting then returned to the agenda and to the standards development tasks. Gary Wiese presented his concept of the tasks that need to be accomplished, and whether they are primarily within the scope of the OEOSC, or SPIE's IRWG. This joint group (acting under the auspices of the OEOSC) will write these standards, and also (working under the auspices of the IRWG) carry out the measurements of the materials. Gary Wiese also presented a summary of how the group could be organized into subcommittees.

5. Follow up on actions from DSS meeting

a. Materials, wavebands, temperatures, and precision

Gary Wiese then went on to provide more information on the scope of the task from his perspective in terms of materials of interest, the wavelength ranges, and the properties that need to be characterized. The documents this group generates will need to contain info on the material properties and also the tests needed to establish the properties of IR materials. Methods for curve fitting of the index data (over temperature) is not even standardized in the visible. NASA's curve fitting procedure captures wavelength and temperature. We are unsure of what sampling of the data is used and where it was established. Inclusions measurements will require developing an instrument, unless we accept what already exists. Schott has built their own measurement system, but it does not resolve inclusions to the level Gary Wiese has requested. The NASA refractometer only goes out to 5 microns and we would like to measure out to 12 microns. We would also like to increase the temperature range to include cryogenic temperatures to satisfy some users of the data. The plan is to develop a schedule, procure material, develop a sampling method and write the statement of work for each of the materials and tests.

A question was asked concerning surface structure and micro structure. Gary's interest is in bulk properties, but we can consider this.

Another question was about birefringence, this would require developing another instrument. It was mentioned that John Burnett at NIST can do birefringence but the instrument would need to be modified for the IR. A show of hands indicated that about half a dozen from the 30 were interested in birefringence. The materials would need to be tested to some extreme mechanical environment to fully establish the stress coefficient.

The developed standards would also have to explain how to make the measurements and establish traceability for qualification.

There was some discussion of where to hold the next meeting. Currently OEOSC meets at PW and O&P. Work will be done and document drafts will be written between meetings and the meetings will be used to review them and establish action items for the next review. The subcommittees Gary Wiese views necessary are: test plans, instruments, sampling, procurement/specifications, and funding. We need to establish what are the IR materials of interest, how to define the class of materials, the wavelength band of interest, the temperature range (the interest level in cryogenics seems minimal with perhaps not all the materials being tested there, however it seemed that most of them were of interest). There is a need to evaluate scattering in some sampling method. Should we consider polymer materials in this IR range? The Schott company indicated that they do handle chalcogenide glasses. And we will need to prioritize these tasks.

We also need suggested precision levels for index, dispersion dn/dt, absorption coefficient, homogeneity and minimum inclusion size. Perhaps also include thermal expansion coefficient as part of the dn/dt. It was mentioned that specific gravity numbers are known for the materials and should these be included in the specifications? Since physical properties are relatively well documented, it will probably not be necessary to do much to standardize them.

While most of these standards will need to be developed by the committee, there are some standards which already exist. Bill Royall mentioned that ISO 20473 defines spectral band, including IR bands. Since it is maintained by TC172, we have access to it, and may be able to modify it to add additional bands of interest, such as SWIR 1, SWIR 2, etc.

In addition, the Military standard for optical glass, MIL-G-174B can be used as a starting point for materials properties.

b. NIST role

The next speaker was Leonard Hanssen from NIST. He indicated that the existing refractometer can be extended to the IR for about \$20 - \$30K. And that this instrument would likely meet the needs of the specification. The NIST Precision Refractometer is managed by John Burnett and he would have the details of its cost and operations procedure. It is an all reflective instrument and the measurements are usually done at room temperature, but it is possible to upgrade to the temperature range required. It can also make the dn/dT measurements, reflectivity, transmittance, and emittance of all types of materials for 1 to 20 microns. He implied that NIST has already begun to plan the upgrade of this instrument, it should have the capabilities we need in 6 months to a year.

Leonard continued to describe the services available from NIST. They can supply standard reference material, calibration services, a measurement assurance program, standard reference data, conduct round robins and organize instrumentation workshops. They can supply samples for purchase (there is some internal funding for some of these), there is also tested material available for loan for characterizing others instruments and for monitoring test capability over time.

The calibration services include custom sample measurement, characterization for a fee, (some funds may be necessary for setup charges). The calibration services have typically higher fees than other approaches.

NIST has a Measurement Assurance Program (MAP) where NIST maintains a sample and loans to a customer, this approach is lower cost.

NIST performs Round Robins and inter comparisons of materials from suppliers. This requires more effort as it involves multiple companies and materials.

A question was asked about the homogeneity measurement concerning beam size, the best guess was that a 6" diameter would like be the maximum needed.

A question was asked if there was an accreditation process offered by NIST, the answer was yes, but it is managed outside the measurement lab. There is a Materials Voluntary Lab Accreditation Program (MVLAP). This process is not free, but there still is interest in it.

NIST asked the audience to determine which process would be of most interest to them. This will need to be resolved by consensus. But the current interest seems to be in a Round Robin set of measurements and the sample loan calibration process.

6. Materials measurement tasks (taken out of order)

At this point Gary Wiese described the previous Round Robin of materials: ZnSe was measured from vis to LWIR, fused Si was measured from UV to MWIR. 3 labs participated and room temperature measurement only were made. The results were fit to a 3 term Sellmeier equation.   Gary presented drawings with specifications on the prisms that were made and tolerances on the angles and surface finish. The data was presented as the Sellmeier terms or tabulated and the measurement error level was established. Most of the variation in the data occurs near the visible edge. Conclusions from this study were that: there is good repeatability in index and dn/dT.

To repeat this on a larger scale for the materials and data ranges of interest will require planning, coordination among companies and Personnel as well as equipment and fabrication costs. We would need to establish the number of samples, labs involved, hoe to collect the data, the types of tests and then qualify suppliers to the established standards. Other items include measurement and sampling protocols as well as tracking all the data.

The material sampling involves determining the sample location (from the boule), size and configuration. For attenuation we also have to establish the thickness. Two thicknesses were suggested to be tested to correct for spectral noise, 5mm and either 10 or 20 mm.

Gary Wiese said he estimated (based on 2 year old data) that there might be 160 samples at a cost of $4500 per sample coming to a total around $1M. Some in the audience thought that the costs could be less for both samples and fabrication as some samples could be donated for free and fab costs have come down.

The data also has to be integrated into lens design software and those companies should participate in determining the best method to do this.

7. Standards development tasks

Dave Aikens spoke next. He is the executive director of OEOSC which administers ASC OP and the ISO TAG to TC172. ASC OP is the accredited standards writing body for the US in the area of optics and optical instruments. They publish standards for the US optics industry and write the documented parts of the standards involving tests.

Dave Aikens asked if there are internal process documents that are currently used in industry that we could use as a first cut for some of the required standards on testing. If anyone has an internal document that could be used by the group, they should send them to him. Dave Aikens then presented his list of standards that will need to exist.

The first was characterization of IR materials and presentation of data, such as index and dn/dT. There is an ISO standard, ISO 11382, which covers this, and may be adequate as is There is also an ISO standard (ISO 17328) for refractive index measurement which is currently in process. Absorption and transmission are covered by ISO 11551 and 15368, respectively. Not sure about bubbles and inclusions but homogeneity might be covered by ISO 17411. Also not sure about stress birefringence. The definition of spectral bands exists in ISO 20473. These may not all be applicable to our needs, but they are useful starting points.

We will need definitions on material properties as specifications and grades as well as how to incorporate that information into libraries for design codes. Mil standard 11382 shows examples of how to put this info on drawings.

Dave Aikens then went through a list of subcommittees and asked for volunteers to start as leaders for each of these topics.

The subcommittees and people who volunteered are:

ISO 17328 (review and study)	– Eric Stover at M3 measurements
dn/dT	– Leonard Hanssen for John Burnett at NIST
Absorption/transmission	– Leonard Hanssen at NIST
Bulk inclusions instrumentation	– Nathan Carlie at Schott USA
Homogeneity and Stress	– Nathan Carlie at Schott USA
Stress Birefringence	– Nathan Carlie at Schott USA and
	– Adam Phenis at SAIC
Spectral Bands	– Gary Weise at LMCO
Material library for design codes	– Dave Hasenauer at Synopsis
Materials specifications and Grades	– Hal Johnson at HJOL and Dave Aikens

8. Time and place for next meeting.

The final discussion concerned the next meeting. It seemed that Photonics West may be a good choice, but DSS is probably better, and is soon after PW. It was felt that the time after the exhibit was better than before, but that some time should be left for tear down. Gary Wiese will discuss the options with Dave Aikens and SPIE and announce the next meeting time and location when available.

9. Adjourn.

A motion was made to adjourn the meeting by Ray Williamson, the motion was seconded by Ed Freniere and it carried. The meeting was adjourned at 16:31.

Recorded by: Ron Scotti
SPIE, Science and Technology Strategist
Acting as secretary.

Edited by D. Aikens for format.