

# Meeting Minutes (Draft 1)

## ASC-OP/TF6 – IR Materials and SPIE IR Materials Working Group

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Wednesday, August 12, 2015

Carlsbad Room, Marriott Marquis San Diego Marina, SPIE O+P Meetings

### **CALL TO ORDER at 8:14 am PDT**

Adam Phenis, Chairman of TF6, called the meeting to order.

### **WELCOME AND INTRODUCTIONS (Agenda Item 1)**

Adam Phenis, ASML-Cymer	David Hasenauer, Synopsis
John Burnett, NIST	Allen Krisiloff, Triptar
Ed Freniere, Lambda Research	Erik Stover, M <sup>3</sup> Measurements
Doug Leviton, Leviton Metrology Solutions	Manuel Quijada, NASA-GSFC
Kevin Miller, NASA-GSFC	Chris Karp, Reynard

### **SCRIBE (Agenda Item 1)**

Allen Krisiloff

### **ADOPTION OF AGENDA (Agenda Item 2)**

Motion by John B. Second by David H. Passed 100%

### **APPROVAL OF MINUTES FROM APRIL MEETING (Agenda Item 3)**

Motion by Allen K. Second by Ed F. Passed 100%

### **PILOT INDEX PROJECT (Agenda Item 4a), John Burnett, NIST**

#### **Overview**

John is still cross-checking the numbers on Ge. He is leery of publicizing results before having completely reviewed the data. He has had to make retractions in the past; he wants to avoid that now!

This project is funded for the remainder of the year.

John reviewed the high resolution refractometer and the measurement protocol used for the pilot study:

Method is consistent with ISO TC172 SC3 N223, Test method for IR index.

$dn/dT$  measured over 15 to 25 degrees centigrade.

Nitrogen purged because Oxygen ( $H_2O$  and  $CO_2$ ) exhibits problematic absorption bands in the IR.

Black body used as light source; Spectral or laser line sources used for calibration of the monochromator.

Control temperature by mixing liquids of two different temperatures. Calibrated with triple point of water. Melting point of gallium. Good for 1 millikelvin absolute accuracy.

Error bars sensitive to surface figure. Increasing size of sample is not significantly advantageous.

Schedule for next samples might change a little.

#### **Size of the original boules and samples**

Samples are about 1.5 inches along a face. Size of original boules are not known. Some samples were cut from near the axis of a boule, others from near the outer circumference.

It was noted that resistivity can change from the top to the bottom of a boule. This pilot study may allow us to evaluate the correlation between resistivity and index.

#### **Homogeneity of the Index of the original boules**

Quality grades for the Pilot Study are “typical”. We don’t know the homogeneity of the boules from which the samples were taken. Homogeneity is more of a problem in amorphous materials like fused silica because they are more sensitive to manufacturing recipes than are crystalline materials like  $CaF_2$ .

Doug has seen  $4E-4$  variations in fused silica from one family of products to another. John has seen  $1E-6$  variations in lithographic grade fused silica.

Crystalline materials are less sensitive to manufacturing recipe but suffer from some unique issues, for example, meandering of the crystal axis in a single boule.

Polycrystalline materials, like polycrystalline  $BaF_2$ , share some quality challenges with amorphous materials.

#### **$dn/dT$**

The CHARMS machine at NASA Goddard is designed to measure down to cryogenic temperatures, which is required for astronomical applications, but John’s machine at

NIST is not yet ready for such a wide range of temperature. The pilot study may have some  $dn/dT$  measures over short ranges.

Thermal issues can be very difficult to assess or control. Temperature can usually be controlled better in gas-filled chambers because thermal conductivity through the gas helps equilibrate the system. In general, changing the temperature of the refractometer is very tricky. Many things are different at different temperatures, and must be well understood in order to achieve the Pilot Study's goal of exceeding  $1E-6$  accuracy.

Some customers ask for temperature sensitivity of Sellmeier coefficients to use in their design software. It is important to deliver the results of the Pilot Study with respect to standard air and at a specific temperature. This will allow easy entry into software libraries. Some manufacturers report data at 25 degrees C; others at 22 or 20 degrees C.

John performs his own calibrations of temperature probes to reach accuracies of one millikelvin. He trusts a vendor's calibration only for a very short period of time.

### **Measurement of Deviation Angle**

John uses the "double minimum deviation" technique to reduce systematic errors in the measurement of the deviation angle. He takes a measurement then rotates the prism around to use a different part of the encoder for a second measurement. He can drive contributions from angle measurement error down to below  $1E-7$ .

Some groups use interferometric methods to reduce angle errors; others use an undeviated beam to establish a stable fiducial angle. These methods seem to be very good, but their accuracy is not known to below  $1E-7$ .

### **Wavelength**

John: with regard to calibration of the wavelength, there are very few long wavelength lines in the infrared.  $CO_2$  laser is really good. There are some good ones in the 2 micron to 3 micron range, and then between 9 and 11 microns. I calibrate the monochromator with the  $CO_2$ . For good signal to noise ratio, we need a grating blazed for the wavelength. Otherwise, we must open slit too wide. I prefer to measure many wavelengths to develop the Sellmeier equation. I use two gratings.

John will investigate the use of the 3.39 micron HeNe line, too.

The software libraries need actual index measurements at fiduciary wavelengths and temperatures so that so as to confirm the accuracy of the dispersion coefficients and the implementation of the dispersion equations.

### **Accuracy versus Precision in General**

John: repeatable (high precision) does not mean low systematic error (high accuracy). In published work, I always wonder about the real systematic errors. I see systematic drift in published studies of the same material over time.

John: Remember that our mission at NIST, and my objective for this pilot study, is to achieve extraordinary, unassailable accuracy. For index, that means beyond the sixth place if at all possible. Then other techniques that are faster and cheaper, but less accurate, can be calibrated with confidence.

### **Schedule and Goals**

Eric: This high accuracy pilot study may uncover the problems that we don't even know exist.

John: maybe we measure in phases, passes. This pilot study is a first pass, exploratory phase. A second phase can zero in on specific issues.

John: I should have completed my review and cross-check of my data for Ge in Sept-Oct.

John: longer term plans. Trying to correlate slow, high accuracy methods with lower accuracy but faster methods. For example, prism coupling techniques.

John: And later, we can move into the cryogenic range.

## **STATUS OF SAMPLES (Agenda Item 4b), Adam Phenis, Cymer/ASML**

### **Overview**

Some suppliers have donated only windows, no prisms.

Some other suppliers are still willing to donate samples, but are late. They tend to be materials less prevalent in IR applications and are scheduled later in the pilot study.

Main goal for the pilot study is to determine the best structure for a future, larger study.

### **Cleartran**

DOW may be closing its Cleartran facility.

### **ZnSe**

Erik will send out ZnSe samples.

### **ZnS**

Adam: We don't have ZnS samples.

## **BaF2**

John: Si or CaF2 great materials for this pilot study. BaF2 less so.

Eric: BaF2 is more hydroscopic, temperature sensitive. CaF2 much more popular by 10x.

## **Spinel**

Spinel would be nice for later in the study. It is used for windows and domes. Polycrystalline. Also known as Cermat. No cleaving. Strong.

## **Si**

Si is also very important for IR applications. However, there are many different production methods and different families of products. Optical properties will be vary.

## **IRG26**

IRG26 is moldable. An important industry is automotive.

## **SOFTWARE UPDATE (Impromptu Agenda Item), Dave Hasenauer, Synopsis, Product Manager for Code V**

### **Overview**

Synopsis, Lambda Research, and other software houses are all ready to incorporate the new data into IR material libraries.

There are several different dispersion equations at our disposal. We need coefficients for the all or several that are deemed appropriate for the IR bands. We will need measured indices at fiduciary wavelengths for error checking.

Here is a comprehensive list of material properties that seem to be important to IR designers:

- bulk scattering for illumination calculations
- Birefringence coefficients for both ordinary and extra-ordinary directions
- Fluorescence coefficients
- $dn/dT$  coefficients
- CTE
- Specific gravity
- Intrinsic resistivity
- Stress birefringence is a problem for current products

Data exchange formats must be determined. Straightforward, but unsettled at this time.

## **Discussion**

Doug: Our customer base for the CHARM measurements is astronomy. Properties at cryogenic temperatures are important for space based instruments. They would be happy to provide CTE. Measurements from CHARM are published in SPIE proceedings.

Erik: Many groups have their own custom libraries.

Adam: It would be really nice to officially standardize an XML format for the library.

## **STATUS OF STANDARDS (Agenda Item 4c), Allen Krisiloff, OEOSC**

### **Overview**

IR Standards are under development in both the domestic ASC OP and the international ISO.

OP1.007, IR Spectral Bands, was recently rejected on the OP level primarily as the result of objections to the proposed definition of IR Abbe values. Abbe values were first defined in the VIS region and are functions of three independent variables: the high and low wavelength boundaries for the Abbe band, and a third intermediate wavelength. All three wavelengths correspond to a relevant emission line.

The proposed definitions for IR Abbe values deviated from this basic philosophy. They were defined as functions of just two independent variables: the high and low wavelength boundaries. The intermediate wavelength was proposed to be the center of the band.

A new draft will be circulated in September with a definition for the IR Abbe values that is consistent with the traditional, visible definition based on three independent variables.

OP1.008, Sampling Protocol, passed on the task force level. It will be circulated to the entire OP in September.

Several IR materials standards that define methods of measurement are under development at the international level, TC172, SC3.

### **Discussion**

OEOSC adheres to ANSI rules regarding ballots and maintains its membership in ANSI because ANSI is the only official American conduit to the ISO.

Nathan Carlie might have additional insight into the definition of Abbe values.

To vote in the balloting of OP1.007 or OP1.008, an organization must register with the OEOSC. For approval at the OP level, ANSI requires a simple majority of voting

members to vote YES, and a 2/3 super majority of submitted ballots. The more registrations from organizations with an interest in IR materials, the easier it is to develop and approve IR standards.

Any schema, XML or otherwise, that we standardize for a library format, could be submitted as a proposed ISO standard, too.

Several measurement techniques can be included in a standard to maintain flexibility, but each one must pass muster as scientifically and commercially valid.

**NEXT MEETING (Agenda Item 6)**

John moved to meet face-to-face at DSS, April 2016, in Baltimore. Erik seconded. Passed 100%.

Between now and then, a webinar or teleconference meeting can be called by Adam as Chairman.

**ADJOURNED Closed 10:36 am**

Allen moved to adjourn. John provided the second. Passed 100%

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**ACTION ITEMS**

**John Burnett:** continue the Pilot Study. Ge results expected in October. He will then start on the next set of samples.

**Allen Krisiloff:** Circulate next round of Standards in September.

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