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Enter TCM and Associated Equipment into Stage One Mockup

Nuclear Technology Research and Development

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SUMMARY

The scope of the FY17 milestone involved entering the TCM into stage one mockup at the Materials and Fuels Complex (MFC) and completing initial testing. While the TCM head was designed and partially fabricated in FY16, the ancillary equipment and software for standalone operation at the IMCL still required development. The tasks required to prepare the TCM for stage one mockup included: 1 – develop cell feedthroughs for both optical and electrical signals, 2 – design and construction of a film thickness monitor, 3 – drafting an equipment qualification plan, 4 – develop an integrated equipment rack to house lasers and instrumentation, and 5 – develop coding instrumentation software for controlling the TCM.

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ACRONYMS

EQP	Equipment Qualification Plan
IMCL	Irradiated Materials Characterization Laboratory
INL	Idaho National Laboratory
IRC	INL Research Center
MFC	Materials and Fuels Complex
TCM	Thermal Conductivity Microscope

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ENTER TCM AND ASSOCIATED EQUIPMENT INTO STAGE ONE MOCKUP

1. INTRODUCTION

The Thermal Conductivity Microscope (TCM) is an instrument designed to measure the thermal properties of irradiated samples. It is being developed for installation in the Irradiated Materials Characterization Laboratory (IMCL) at the Materials and Fuels Complex (MFC), which is part of the Idaho National Laboratory (INL). The TCM simultaneously measures the thermal diffusivity and conductivity using modulated thermoreflectance. This measurement approach involves measuring the temperature field spatial profile of samples excited by an amplitude modulated, continuous-wave laser beam. A thin gold film is applied to the samples to ensure strong optical absorption and to establish a second boundary condition that introduces an expression containing the substrate thermal conductivity. The diffusivity and conductivity are obtained by comparing the measured phase profile of the temperature field to a continuum based model. The TCM has been designed to operate in a radiation hot cell environment. It can be controlled remotely via the software interface and sample loading is compatible with hot cell manipulators.

2. FY17 MILESTONE

The scope of the FY17 milestone involved entering the TCM into stage one mockup at the MFC and completing initial testing. While the TCM head, which is the system component that makes the measurements, was designed and partially fabricated in FY16, the ancillary equipment and software for standalone operation at the IMCL still required development. The tasks required to prepare the TCM for stage one mockup included: 1 – develop cell feedthroughs for both optical and electrical signals, 2 – design and construction of a film thickness monitor, 3 – drafting an Equipment Qualification Plan (EQP), 4 – develop an integrated equipment rack to house lasers and instrumentation, and 5 – develop coding instrumentation software for controlling the TCM.

2.1 Design and Construction of Cell Feedthroughs for both Optical and Electrical Signals

The TCM head was designed for installation in a glove box at the IMCL. Lasers, laser controllers, stage controllers and other ancillary instruments and power supplies are located outside the glove box. Sealed feedthroughs to transmit the optical laser signals and electrical signals through the glove box wall required development. It was determined that the feedthroughs would be incorporated into standard vacuum fittings to meet leak rate requirements and for ease of installation and replacement if necessary. A vendor was identified who could provide the custom optical fiber feedthroughs. Vendors were also identified who could provide the electrical feedthroughs in standard vacuum flanges. The feedthroughs were procured for initial testing. A removable glove box panel incorporating the mating flanges for all of the required feedthroughs has been designed and will be fabricated after feedthrough testing is complete.

2.2 Design and Construction of a film thickness monitor

A unique capability of the TCM is the ability to directly measure thermal conductivity and diffusivity using a single-sided measurement approach. The sample is coated with a thin gold film which ensures strong optical absorption and provides a boundary condition which allows direct determination of the thermal conductivity. The gold film thickness is a required parameter for the analysis. The film thickness is determined by measuring the ratio of light transmitted through a film applied to a transparent surrogate (BK7 optical glass) coated simultaneously with the sample. This measurement is easily made in a laboratory setting, but performing the measurement in a glove box using manipulators required the design and fabrication of a dedicated device. The film thickness monitor was designed to allow a manipulator to easily place the surrogate sample in position and transfer it into a laser beam for transmission measurements. It consists of a tower which directs a fiber coupled laser beam from the top of the tower onto a photodiode in the bottom of the tower. A drawer with a hole for light transmission can be rotated into the beam path using a manipulator friendly handle. With the handle in the load position the drawer is rotated out of the tower so a sample can be placed in position. When the handle is moved to the measurement position the sample is moved into the beam path for the transmission measurement and film thickness determination. The laser beam is contained within the device. The thickness monitor was designed and fabricated and was included in the initial mockup testing.

2.3 Writing an Equipment Qualification Plan

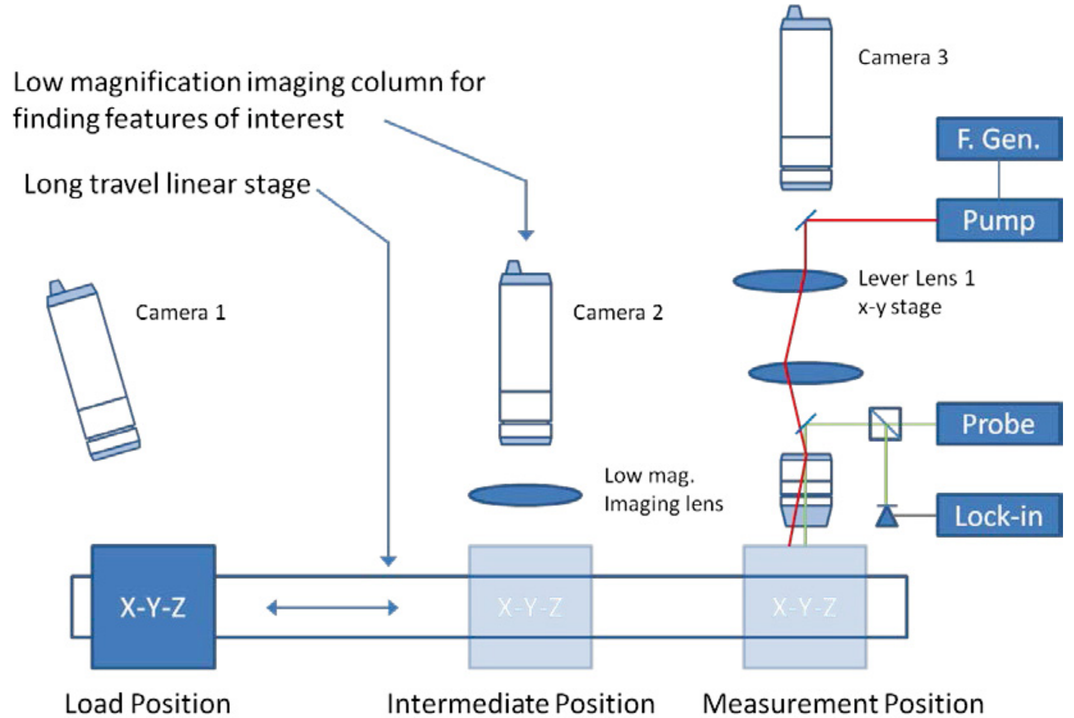
A Remote Equipment Qualification Plan (EQP) for operation of the TCM at the MFC was finalized in June 2017. The functions of the EQP are to provide work control documentation for testing of the TCM at the MFC, provide basic instructions for assembly and operation of the TCM, and provide verification that measurements made using the TCM at the INL Research Center (IRC) laboratory could be duplicated at the MFC mockup shop. The Phase I mockup testing covered by the EQP is an initial requirement prior to the installation of the TCM into a hotcell at IMCL.

2.4 Develop an integrated equipment rack to house lasers and instrumentation

The TCM instrument head which performs the thermal conductivity measurements will ultimately be located in a glove box at the IMCL. In the laboratory environment ancillary support equipment has been located on the optics table next to the TCM or on shelving over the optics table. Operation at the IMCL will require ancillary equipment to be mounted in a rack next to the computer work station located approximately 30 feet from the TCM in the glove box. In addition to mounting equipment in the rack, an existing interface box and a newly developed power/switch box allow computer control of the TCM and thickness monitor. The interface box provides a laser interlock and a convenient device for switching and controlling power and signals. The power/switch box accepts signals from the interface box to control the power to the photoreceivers, cameras, illumination lighting and sample detector in the TCM head. This box also allows the function generator and lock-in amplifier signals to be switched between the TCM and the thickness monitor eliminating the need for duplicate instruments. The sample detector consists of a slot detector which will be used in conjunction with the sample stages to determine sample height so travel limits can be set to prevent sample to objective interference. Interface cabling for the interface box and power/switch box was designed and fabricated and the 30 foot cabling from the instrument rack to the feedthroughs was used in initial qualification testing. A sketch of the TCM head with sample stages and the thickness monitor is shown in Appendix A. A photograph of the equipment rack next to the TCM at the mockup shop is shown in Appendix B.

2.5 Design and Coding Instrumentation Software for Controlling the TCM

The graphical user interface (GUI) developed using MATLAB provides point-and-click control of the Thermal Conductivity Microscope. In this section, an overview of the instruments that are controlled by the GUI is presented. In Fig. 3 a sketch of the instruments that make up the TCM is given. A long travel linear stage is used to move the sample stage from the load position to the measurement position. In the load position the sample is placed on the sample platform. Positioning of the sample platform is performed using a GUI controlled x-y-z stage. Camera 1 provides the operator with a view of the sample stage in the load position. The sample is then moved to an intermediate position. In this position the sample surface is imaged using camera 2 with a low magnification lens allowing the user to more easily locate a region of interest on the sample.



The sample stage is then moved to the measurement position. In this position the sample is brought into focus and the region of interest can be fine tuned. In addition to the x-y-z sample stage, the GUI controls the x and y position of lever lens 1. By moving lever lens 1, the pump beam is scanned relative to fixed probe beam. In this position there are two subroutines that are used for final alignment. First the beams are made coaxial by performing x-y scans. Second the beams are brought into focus by scanning the sample stage in the z direction. While these operations can be performed manually, having them controlled by the computer increases data reproducibility.

A single thermal wave profile is recorded by first moving the pump beam 10 microns from the probe. After waiting a specified amount of time (related to lock-in time constant) the output of the lock-in is recorded and the pump beam is then moved towards the probe beam and the output of the lock-in is recorded again. This process is repeated until a full profile has been recorded at a single frequency. Scans at multiple frequencies are conducted and the data is then fit to a continuum based model to determine the thermal conductivity. The entire process from loading the sample to data analysis can be controlled through the graphical user interface.

3. FY17 RESULTS

Preparations for mockup testing of the TCM were completed in August 2017 and the TCM was delivered to the mock-up shop at the Fuel Conditioning Facility at the Materials and Fuels Complex (MFC) on Sept. 5, 2017. Facility approvals, laser inspections and setup of a temporary laser control area, as required by the EQP were completed the following day.

Remotized mechanical manipulator operations as required in section 5.2.2 of the EQP were then conducted as part of the stage one mockup qualification. The manipulator operators were able to demonstrate the ability to place samples on the thickness monitor tray and operate the lever to move the sample into the measurement position. The sample was also placed on the sample platform of the TCM. Once the sample is placed on the platform, positioning of the sample for TCM scans is accomplished using the control computer. Suggestions from the manipulator operators to improve the ability to remotely place samples included making the sample pocket in the thickness monitor tray slightly deeper to make it easier to determine when the sample is correctly located and adding dark laser etched circles and lines to the thickness monitor tray and the sample platform.

Testing of the TCM in mockup was initiated on Sept 7, 2017. Thickness measurements were made on each of the substrate standards tested in the laboratory. When the gold film is sputtered coated onto the sample, a BK7 standard is coated at the same time and is used for film thickness measurements. The Borofloat 33 was coated alongside the BK7 listed in the table but was measured independently. Results of the thickness measurements are shown in Table 1.

Table 1. Thickness Monitor Gold Film Thickness Measurement Results.

Sample	IRC	MFC
BK7	46 nm	46 nm
Borofloat 33	45 nm	45 nm
CaF2	99 nm	98 nm
ZnSe	176 nm	150 nm

The IRC and MFC thickness measurements are in excellent agreement with the exception of the ZnSe. Losses in the long optical fiber reduce the light intensity at the sample such that films thicker than ~125nm reduce signals to the noise level resulting in poor sensitivity. This issue will be addressed by changing to multimode fibers and a multimode feedthrough to reduce optical losses.

After measuring the film thicknesses, the thermal properties of each sample were measured using the TCM. The BK7 standard that was coated alongside each sample was first analyzed to determine the film properties and the sample was measured 3 times. Twice in the same location and once at a different location. The results are presented in Table 2.

Table 2. TCM Standards Testing Results.

	IRC Measurements			MFC Measurements		
Sample	**Film K (W/m°K)**	**K (W/m°K)**	**D (m2/s)**	**Film K (W/m°K)**	**K (W/m°K)**	**D (m2/s)**
CaF2	157.7	10.659	3.22E-06	164.7	11.5	3.18E-06
—	—	10.822	3.29E-06	—	11.58	3.19E-06
—	—	10.213	3.13E-06	—	11.18	3.13E-06
—	Mean:	10.6	3.2E-06	—	11.4	3.2E-06
ZnSe	166.2	18.07	9.51E-06	171.4	18.95	9.56E-06
—	—	16.9	9.11E-06	—	18.66	9.59E-06
—	—	18	9.21E-06	—	17.82	9.39E-06
—	Mean:	17.7	9.3E-06	—	18.5	9.5E-06

As shown in the table, agreement between measurements taken at IRC vs MFC is quite good. The largest discrepancy is about 7.5% between the mean thermal conductivities of CaF2. The other mean values all agree to better than 5%. The literature values of ZnSe (k = 18 W/m°K, D=1.007e-5 m2/s) are also in good agreement with a maximum variation of 7.5% on the diffusivity. There is greater discrepancy in the literature values of CaF2 (k = 9.71 W/m°K, D=3.44e-6 m2/s) with a maximum discrepancy of about 25% on the conductivity and 7.5% on the diffusivity. A discrepancy in the values when scanning in different directions was also noted. These issues will be addressed as part of the Phase II mockup testing.

4. CONCLUSION

The FY17 milestone of entering the TCM into stage one mockup and complete initial testing was met in early September 2017. The TCM had been delivered to the MFC mockup shop earlier and a temporary laser control area setup as required by the EQP. Sample loading using the remote manipulators was demonstrated and initial testing of calibration standards was completed on Sept 7. Testing results showed good agreement between measurements taken in the IRC and those taken at the MFC mockup shop. The issues identified during testing will be addressed prior to completion of phase two mockup testing. The following appendices provide support documentation of the work completed this year. Appendix A contains a sketch of the TCM head assembly with the stand and sample stages as well as the thickness monitor. A photograph of the TCM in mockup with the equipment rack is shown in Appendix B.

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Appendix A

Sketch of the TCM Head Assembly

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Sketch of the TCM Head Assembly

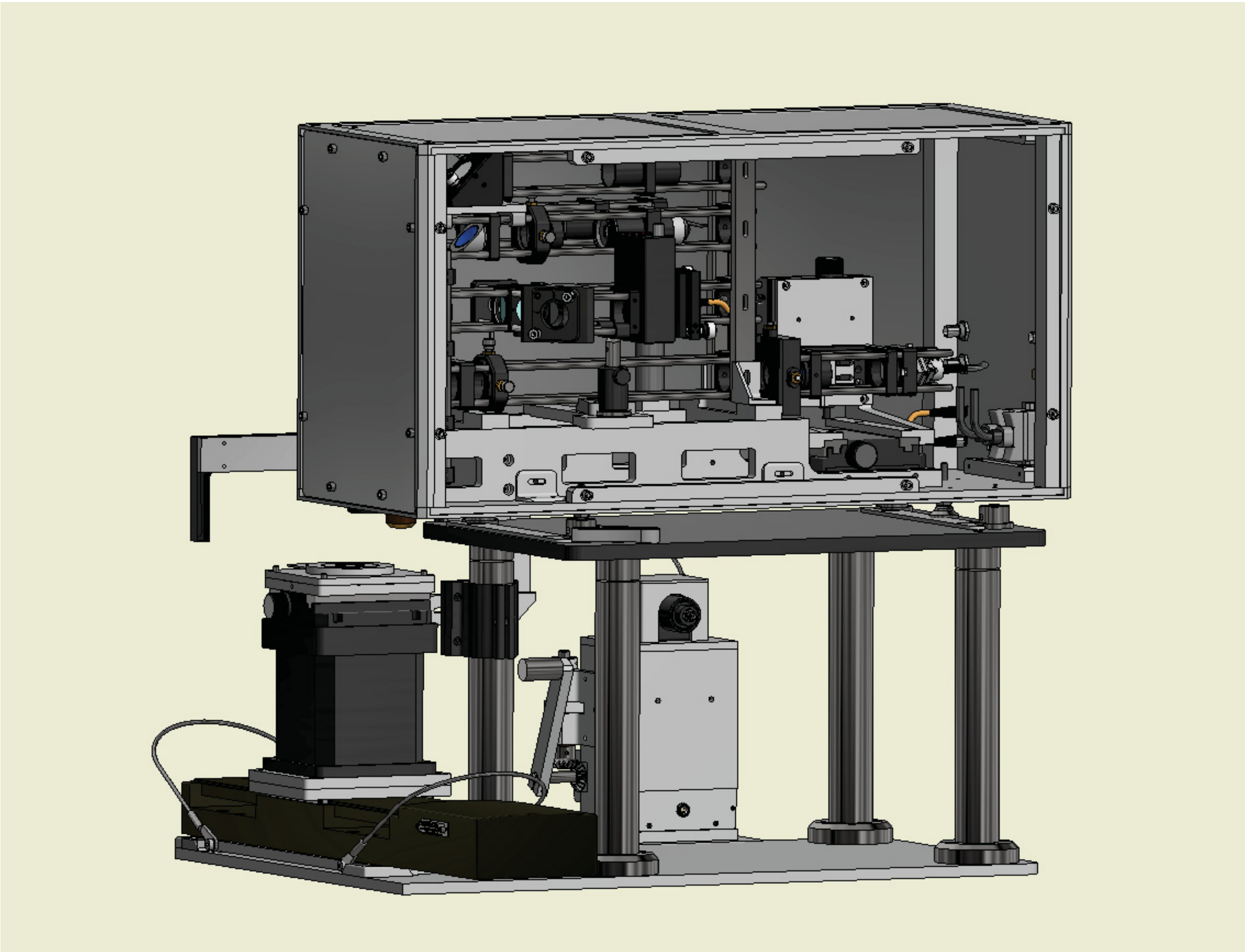

Figure A-1. TCM head assembly showing internal components, sample stages and the thickness monitor.

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Appendix B

Photograph of the TCM at the MFC mockup shop

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Photograph of the TCM at the MFC mockup shop

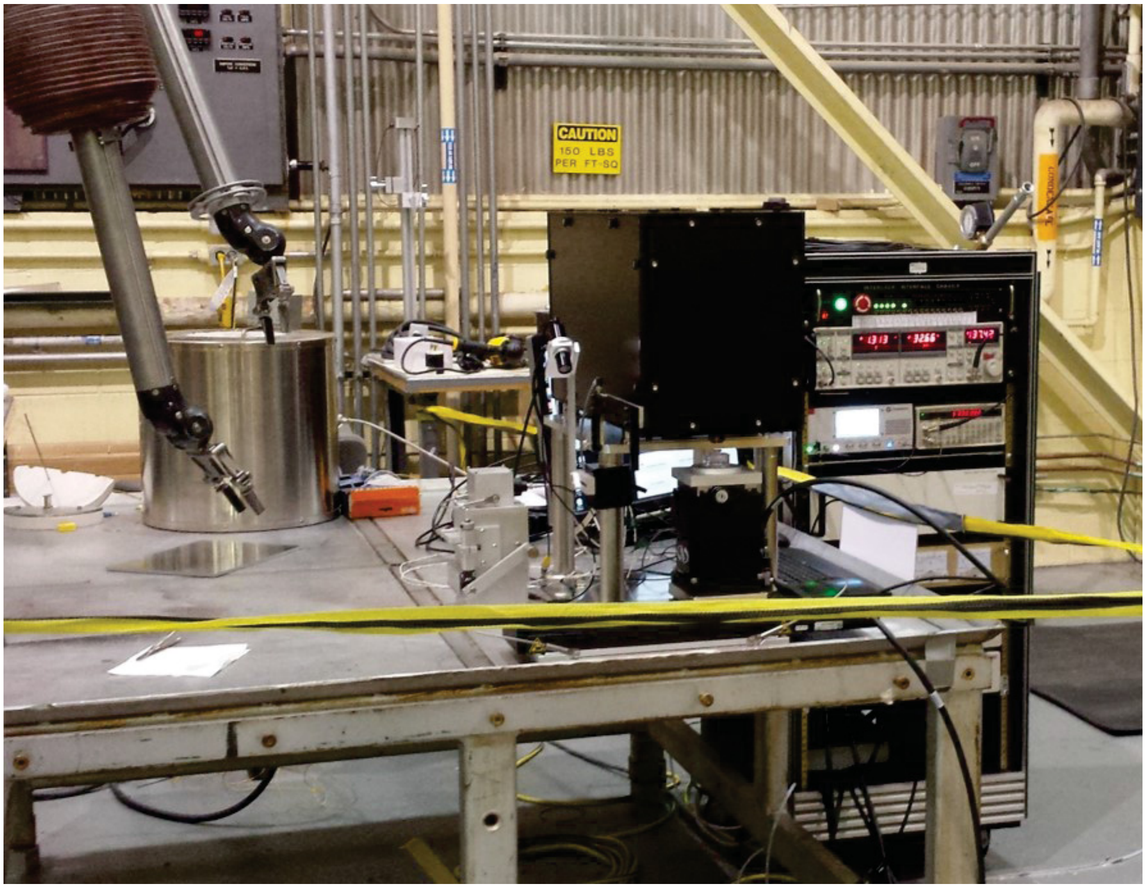

Figure B-1. TCM in mockup testing at MFC.