Hesse Instruments

The Heating Microscope and EMI III Software

Determine the characteristic temperatures according to international standards of ash fusibility

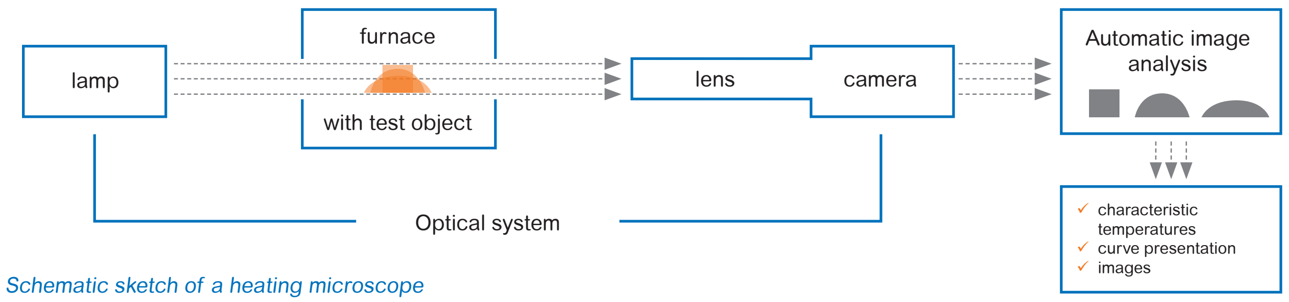
Non-contact, quantitative analysis of the temperature-dependent form and size change

Measuring, evaluating, documenting and managing data – all with EMI III

The Heating Microscope and EMI III Software

The Hesse Instruments heating microscope is a complete test system to easily determine the high temperature characteristics of a wide range of materials. Its method of measurement is based on thermo-optical analysis. The EMI III software controls both the hardware and the software functions; the user can focus entirely on the samples, their analysis and evaluation.

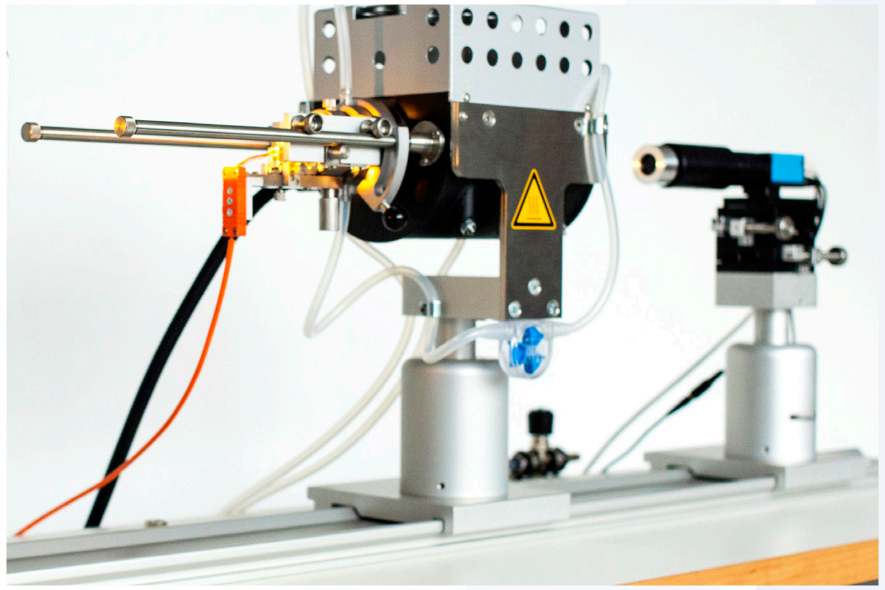
The silhouettes of a test object are continually analyzed, while it is being heated in a small tube furnace: The so-called characteristic temperatures describe the melting behavior of ash according to the standards based on deformation, shrinkage starting, sphere, hemisphere and flow temperature.
These characteristic temperatures relate to geometric shapes that a test object adopts, while it is heated, softens and melts.
The heating microscope EMI III software calculates the characteristic temperatures according to DIN 51730, ISO 540, CEN/TS 15370 and CEN/TR 15404 using fixed algorithms. This is how in a short time precise, reproducible results are obtained independently of the person who performed the measurements.
Dilatometric curves are created based on the quantitative determination of the test object area, height and width. From this can be derived for example: Information on sintering and swelling or even varying direction-dependent shrinking behavior. Wetting behavior of a melting phase on several substrates can also be described via contact angel measurements.
All of the effects that go together with a size and/or shape change of the test object can be proven by the heating microscope and the EMI III software.



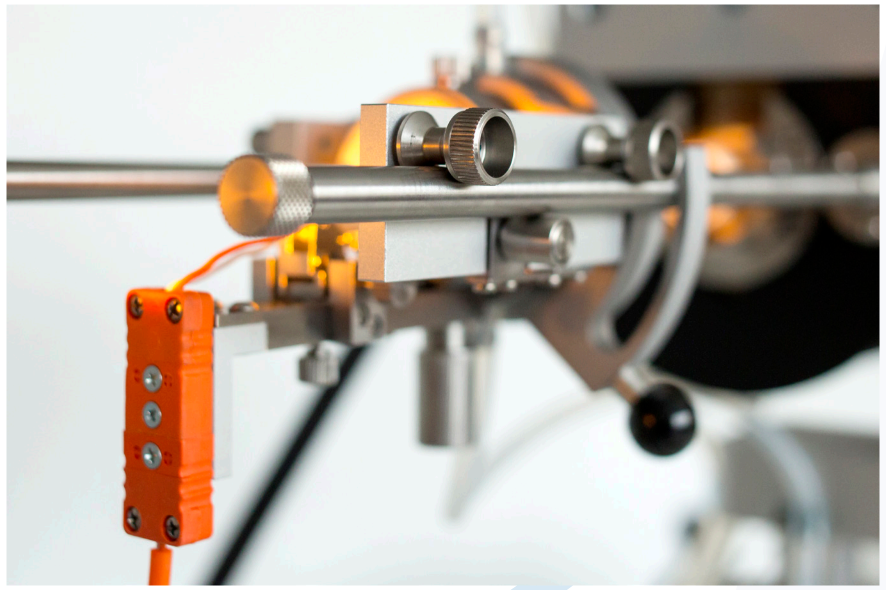
Schematic sketch of a heating microscope

An Optical Bench as the Basic Design

All components of the heating microscope are arranged on an optical bench: this reliably ensures exact alignment of the lamp, furnace and camera, which is essential for precise image analysis. Once set, an optimal specimen position will remain stable, yet can be easily changed by the user to a new position, whenever needed. As each silhouette is analyzed in the direct beam path, no complicated optical components have to be used. Therefore, the entire system is robust and easy-to-service.

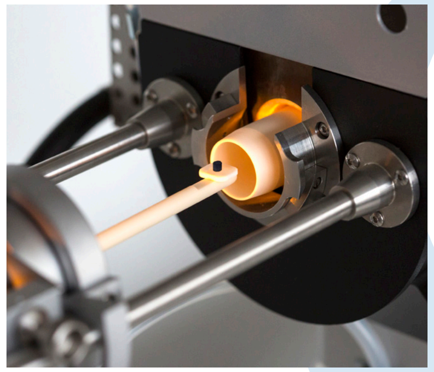


Optical bench



Specimen carriage

Another distinctive element of the heating microscope is its specimen carriage, which is moved on rails that are rigidly attached to the furnace carrier. This unique design enables you to position a specimen on the sample holder outside the (hot) furnace and then to move the sample on the specimen carriage consistently and accurately to its measurement position inside the furnace. At the same time, the furnace is sealed off. As a result, this ingenious design makes sample handling exceptionally easy.

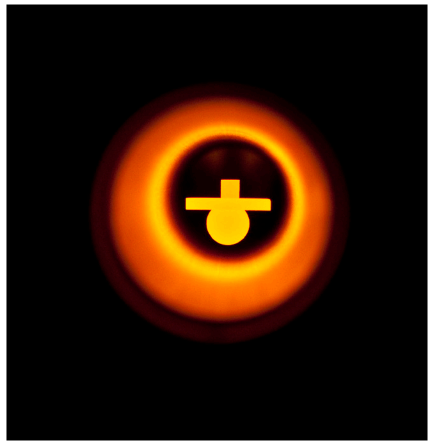


Sample holder

A key feature for accurate and representative measurement of sample temperature is the arrangement of the thermocouple. In the Hesse Instruments heating microscope, this thermocouple is located directly below the specimen in the sample holder. The closed construction of the sample holder ensures optimal three-fold protection of the thermocouple: against mechanical damage, influences from the furnace atmosphere and, above all, against contamination by the sample itself. Over the long term, this construction minimizes error in measurement of the temperatures of specimens – and thus in measurement of their characteristic points.

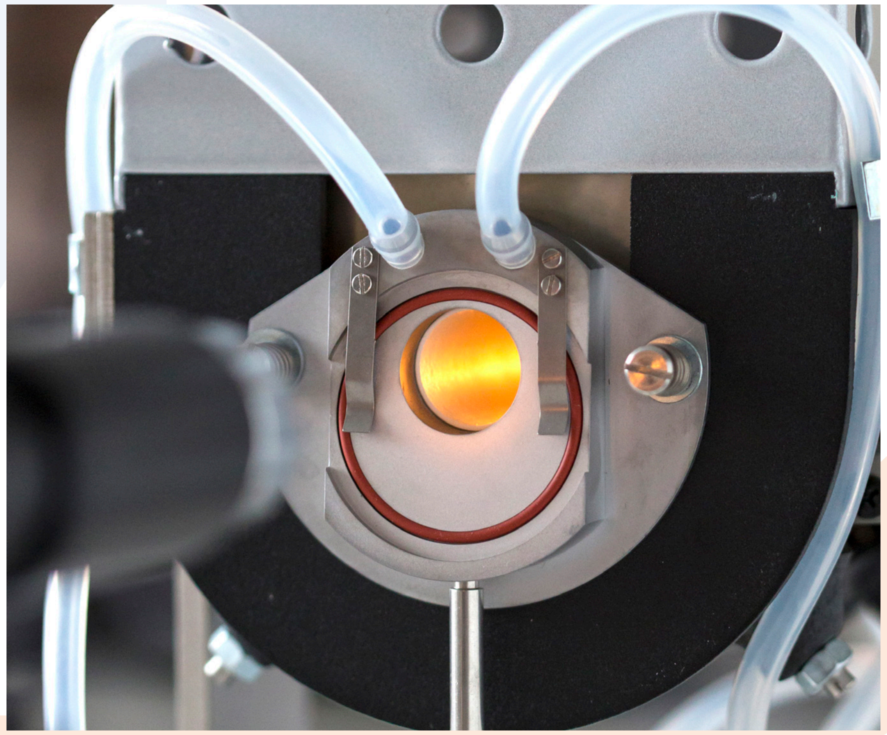
Technically Sophisticated Furnace Systems

Small-scale, compact and technically optimized tube furnaces have been designed for use in the Hesse Instruments heating microscope. Thanks to their highly effective ceramic fiber insulation and advanced heating elements made of molybdenum disilicide ($MoSi_2$), you can operate the heating microscope at high heating rates. For relatively large test series and high numbers of samples, this enables you to measure individual specimens in rapid succession and, therefore, to achieve high sample throughputs.



View into furnance

As the samples are positioned in the confined space of the furnace tube with exceptional accuracy, each sample is exposed to exactly the same temperature field – as a result, repeatability of the measurement results of a test series is better than that in relatively large furnaces and better than that defined in the respective standards.



View to the furnance

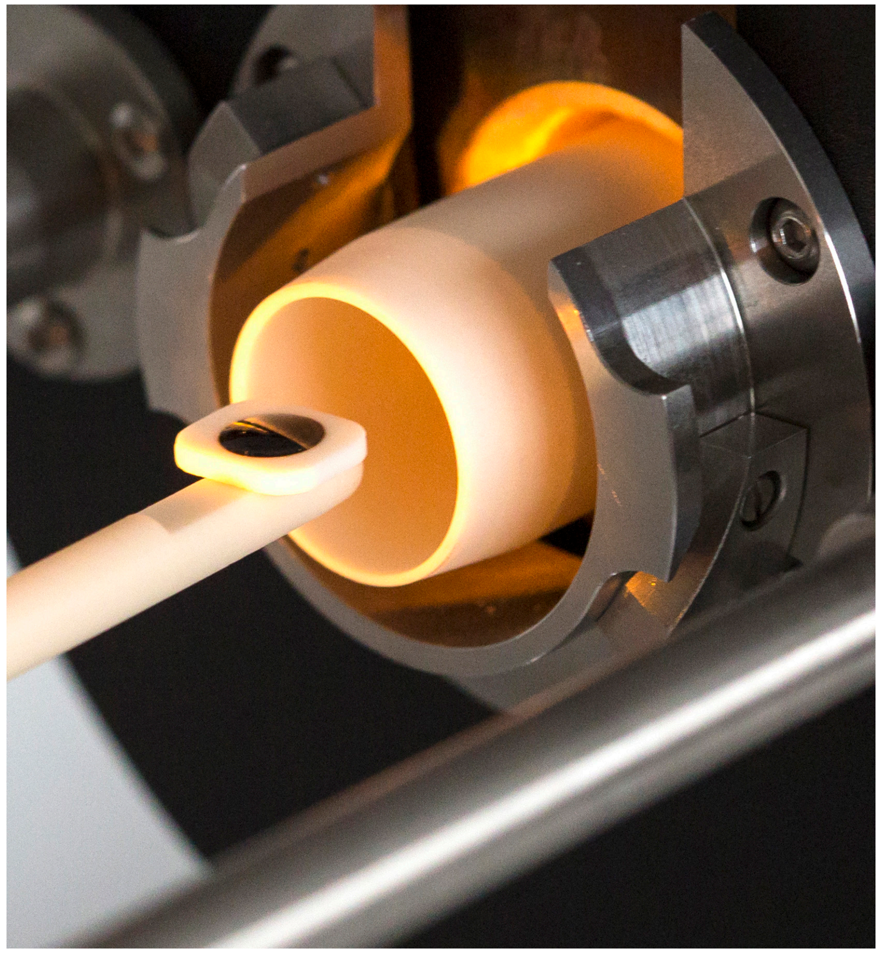
Furnace control unit

To offer suitable solutions for different applications with respect to the heating rate and operating temperature, Hesse Instruments offers you a choice of furnace models for integration into the entire system.

Setting of defined furnace atmospheres is, of course, possible, as required in the standards for the determination of the ash fusibility; also a simple vacuum can be created. To meet requirements beyond these, Hesse Instruments will custom-design the equipment solution just right for you.

The technically sophisticated furnace control unit features an elaborate safety design: heating element temperature, heating current and water flow rate for the furnace closures are continuously monitored.

The furnace tube made of dense-sintered aluminum oxide can be exchanged in just a few easy steps. Thanks to a well-thought-out safety design, easy-to-exchange furnace tube and the $MoSi_2$ heating element, Hesse Instruments furnaces have an exceptionally long life.



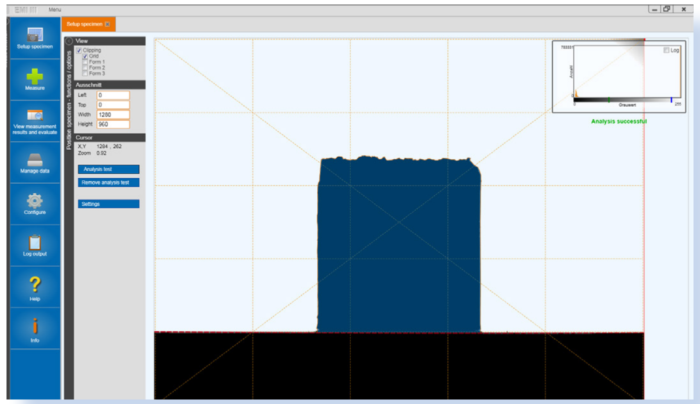
Furnance tube

The Core of the Heating Microscope – the EMI III Software:

The operation of the EMI III software is based on the workflow in the laboratory, which means the software can be used easily and intuitively. The training period is extremely short. Routine applications are done quickly and safely.

As soon as the test object is prepared and placed in the furnace with substrate on the sample holder, EMI III supports any further work with the heating microscope:

Sample setup: EMI III offers auxiliary functions for the alignment of the camera and lens, with which the image settings for the analysis can be optimized.
A successful analysis test confirms the appropriate setting for the image analysis.

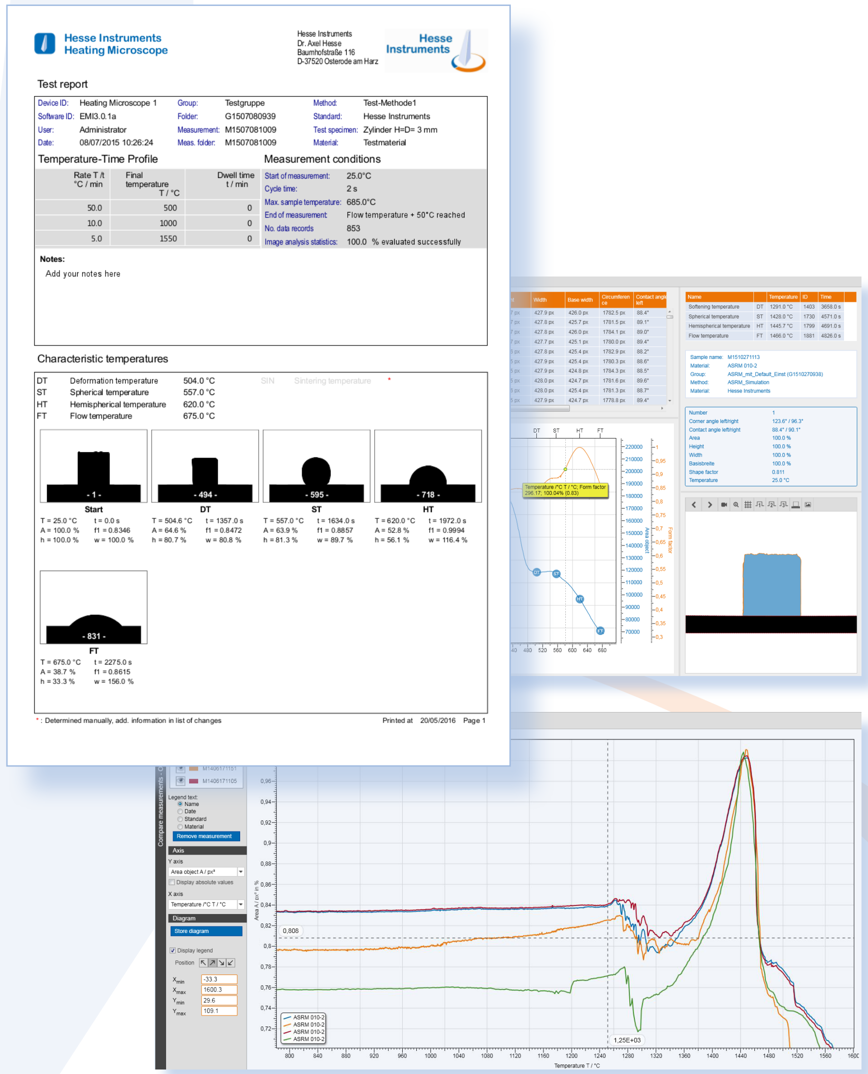


Measurement: Step-by-step guide for the measurement and its documentation of all necessary information, such as the temperature/time profile, the starting temperature or the criterion to finish the measurement. Queries ensure that nothing is forgotten. Routine conditions are recorded in the method storage, the corresponding measurement conditions are in this way at any time quickly available.

When a measurement is started, information about the progress of the measurement is displayed on the screen: in tabular form, in curve form and, of course, images of the test object in real time. During an ongoing measurement most of the features of the software are blocked, since the measurement must be completed without any interruption.

Evaluation: When a measurement has been completed, the results page is displayed with the measurement values, characteristic temperatures, graphical presentation of the results and images. A standardized one-sided summary record can be used as a clear test report. All of the important measurement conditions and results and images of test object silhouettes are documented and a comment in text form can be added.

Alternatively, an individually comprehensive test report can be compiled. This one can contain as many images, graphs, text and data tables as desired. If changes have been made manually, e. g. sintering or deformation temperature was manually set, then this is documented as a change in the test report. The evaluation and documentation of the measurement conditions and results meet all current quality management requirements.



Data management: Data management enables easy archiving and, when required, the recovery of measurement data (including existing evaluations) – also on a second computer. So, regardless of data filters the number of displayed measurements can be controlled. In addition, it is possible to measure in the lab, while evaluating measurements can take place in various workplaces.

Configuration: A user with administrator privileges can manage users and methods, change some program settings and insert a company logo in the test reports. For very special applications parameters of the image analysis can be changed, so that the image analysis can be set more (e.g. for contact angle measurements) or less sensitive (e.g. to easier compare current results with old results from EMI 2.x).

The Most Important Features at a Glance

The heating microscope from Hesse Instruments

Model	EM301-M16	EM301-M17	EM301-R175
Max. furnace temperature	1600 °C	1700 °C	1750 °C
Max. sample temperature	1500 °C	1600 °C	1650 °C
Sample thermocouple	Type S	Type B	Type B
Maximum heating rate	80 K/min up to 1400 °C 50 K/min up to 1600 °C	80 K/min up to 1400 °C 50 K/min up to 1600 °C 10 K/min up to 1700 °C	30 K/min up to 1000 °C 15 K/min up to 1600 °C 10 K/min up to 1750 °C
Heating element	Molybdenum disilicide ($MoSi_2$)	Molybdenum disilicide ($MoSi_2$)	Rhodium (Rh)
Working tube 99.7 % Al_2O_3	Replaceable without tools	Replaceable without tools	Firmly mounted, heating element and working tube are inextricably linked.
Sample thermocouple	Protected against mechanical and chemical influences in an alumina protective tube, positioned directly below the sample.		
Atmospheres	Oxidizing and reducing, inert gas		
Furnace control	Program controller, temperature amplifier, limit temperature monitoring, current limiting		
Specimen carriage	Guided carriage; placing and aligning the samples outside of the furnace.		
Optical components	Halogen direction lamp, CCD camera (resolution: 1280 X 960, format: 1/3"), macro lens, which is tailored to camera and lamp		

Software for heating microscope – EMI III

Software	Operation and control of all functions of the heating microscope.
Sample set up	Optimization of the image setting for automatic image analysis.
Measurement	Performance of the measurements according to user defined criteria, method storage, characteristic temperatures according to DIN 51730, ISO 540, CEN/TS 15370 and CEN/TR 15404.
Evaluation and documentation	One-sided, standardized summary record and brief or individually arranged detailed test report with method description, images, diagrams (representation of several parameters of one measurement or one parameter for multiple measurements), data tables, comments and change tracking.
Data management	Archiving and recovery of data, allows evaluation on the second PC, independent of measuring station in the laboratory.
Configuration	Users with administrator privileges can change individual software settings and add e. g. the company logo.

Resulution, uncertainty of measurement, sample size…

Sample temperature	Resolution 1 K, internal resolution of 0.01 K, standard measurement uncertainty ≤ 5 K
Length	Depending on system settings for hardware and software, achievable length resolution < 0.1 µm, relative resolution better than 0.01 %
Sample material	Powder compacts, drilling cores, samples sawn out of the whole.
Test specimen form	Cylinder, cube and truncated cone according to DIN 51730, ISO 540, CEN/TS 15370 and CEN/TR 15404.
Sample size	Possible sample height approx. 1 ... 6 mm, possible diameter or width approx. 1 ... 8 mm
Sample quantity	Depending on material characteristics (shrinkage, elongation, wetting)
Substrate size (small plate)	Maximum of 14 mm x 16 mm, typically 10 mm x 12 mm; thickness approx. 1 mm

We reserve the right to make technical changes. Latest version June 2016

Hesse Instruments

Hesse Instruments Dr. Axel Hesse
Baumhofstraße 116 • D-37520 Osterode am Harz
Tel. +49 (0) 55 22/75 75 0 • Fax +49 (0) 55 22/76 07 3
sales@hesse-instruments.de • www.hesse-instruments.de