

**NETZSCH**

Differential Scanning Calorimetry



DSC 404 **F1**

*Pegasus*®

Leading Thermal Analysis.

# DSC 404 **F1 Pegasus**<sup>®</sup> – Introduction to the Method

For over 45 years, NETZSCH Analyzing & Testing has been a leading manufacturer of high-performance thermal analysis systems – flexible, sophisticated and technically outstanding. Our customers' wishes and requirements are our guidelines. This, combined with experience and innovation, allows us to consistently set new benchmarks and standards in the field of thermal analysis instrumentation. Our success is a result of the creativity and enthusiasm of our engineers and scientists and our close cooperation with you as our customer.

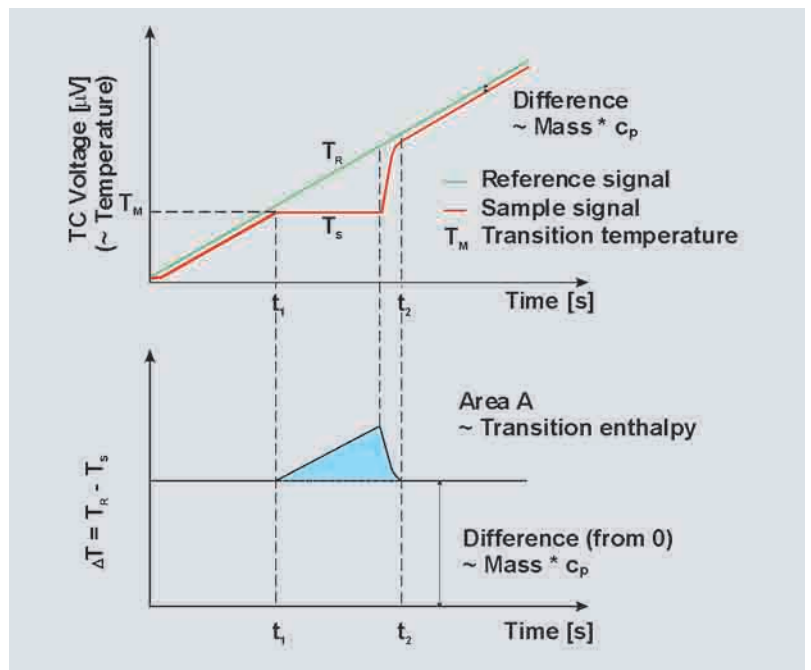


Differential Scanning Calorimetry (DSC) is one of the most frequently employed Thermal Analysis methods. It can be used to analyze nearly all energetic effects occurring in a solid or liquid during thermal treatment. The NETZSCH DSC 404 **F1 Pegasus**<sup>®</sup> operates according to the heat flux principle. Using this method, a sample and a reference are subjected to a controlled temperature program (heating, cooling or isothermal). The actual measured properties are the temperature of the sample and the temperature difference between sample and reference. From the raw data signals, the heat flow difference between sample and reference can be determined. The DSC 404 **F1 Pegasus**<sup>®</sup> meets nearly all respective instrument and application standards, including: ISO 11357, ASTM E 967, ASTM E 968, ASTM E 793, ASTM D 3895, ASTM D 3417, ASTM D 3418, DIN 51004, DIN 51007, DIN 53765.

Analysis possibilities:

- Melting/crystallization behavior
- Solid-solid transitions
- Polymorphism
- Degree of crystallinity
- Glass transitions
- Cross-linking reactions
- Oxidative stability
- Purity determination
- Specific heat
- *Thermokinetics*

Signal generation in a heat flux DSC



# Differential Scanning Calorimeter DSC 404 **F1** Pegasus®

The NETZSCH DSC 404 **F1** Pegasus® allows determination of caloric effects (transformation temperatures and enthalpies) up to the highest of temperatures with outstanding reliability, resolution and accuracy. The high vacuum tight system offers true DSC performance over an unrivaled temperature range. Metal housed mass flow controllers and stainless steel gas tubings make the system an ideal tool for measurements on materials sensitive to oxidation. Enthalpy changes and the specific heat can be analyzed at unmatched levels of accuracy.

The DSC 404 **F1** Pegasus® comprises a heat flux DSC system capable of doing even specific heat measurements. The system can operate from -150°C to 2000°C using various interchangeable sensors and fur-

naces. The optional ASC system improves sample throughput and allows time effective operation.

The DSC 404 **F1** Pegasus® is designed with unique furnaces offering a highly homogeneous heat flow from all sides to the centrally located DSC sensor. The sensors combine optimum sensitivity, a short time constant and a highly stable and reproducible baseline. Therefore, determinations such as phase transition temperatures and enthalpies can be made at lowest uncertainty levels. The specific heat can be measured up to 1500°C at accuracies better than 2.5% for most materials. The various DSC sensors

offer true DSC performance over an unmatched temperature range (-150°C ... 1750°C). DTA sensors can cover the complete temperature range up to 2000°C. The high vacuum tight design, along with high-resolution, metal housed MFC systems and various interchangeable furnaces and sensors, make the system to an ideal tool for top-level DSC research in academia and industry.

The different possible furnace hoists for one or two furnaces, the optional automatic sample changer for up to 20 samples, the different selectable pumps, and a great variety of crucible types allow the system to accommodate nearly all possible materials and application ranges. It is an ideal base system which can easily be optimized and adjusted according to your needs.



# DSC 404 **F1 Pegasus**<sup>®</sup> – Groundbreaking Technology

## Vacuum tight design – optimum atmosphere control

The DSC 404 **F1 Pegasus**<sup>®</sup> stands out as a first rate vacuum tight high-temperature DSC. Practically every component is constructed to fulfill the requirements of high-vacuum and high-purity gas applications. Various pump systems, such as rotary and turbo molecular pumps, are available to evacuate the system down to 10<sup>-4</sup> mbar. Automatic evacuation is of course possible, as is back-filling with

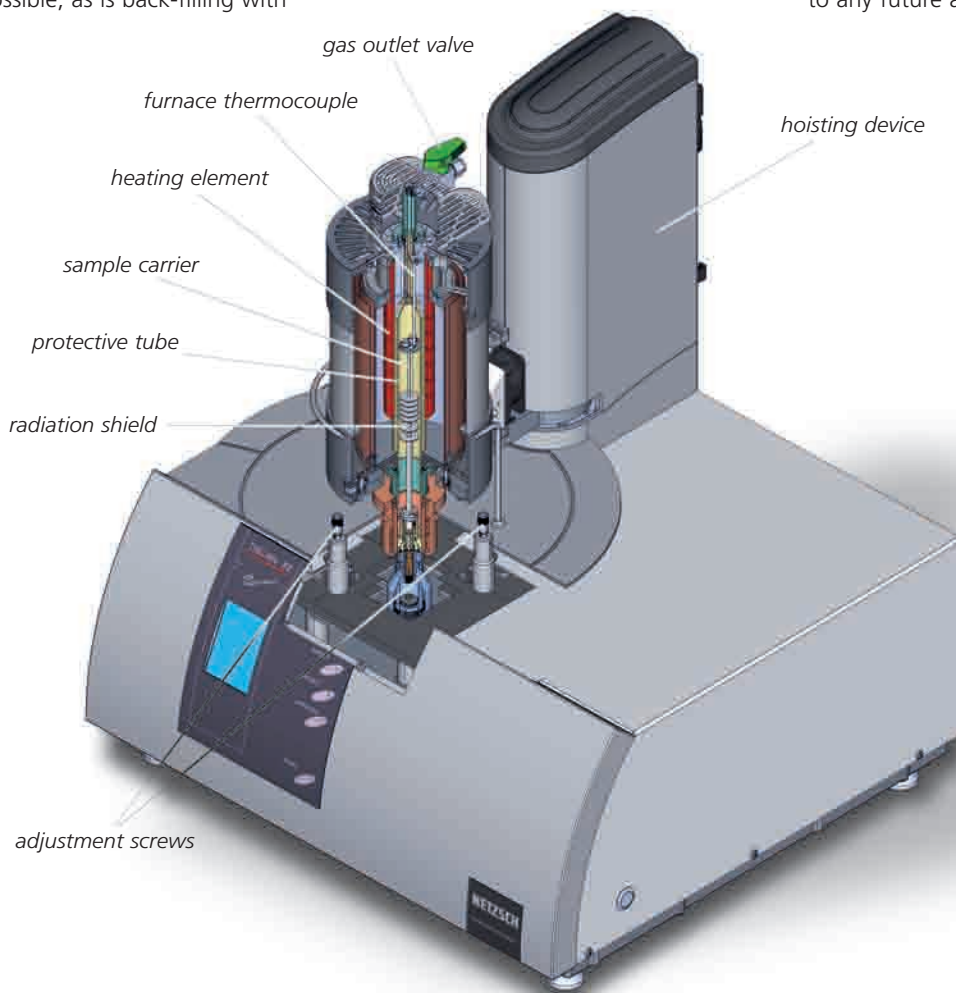
various kinds of purge gases. The OTS<sup>™</sup> accessory can be used to reduce the oxygen concentration at the sample below 1 ppm.

## Mass flow controllers

The system comes with integrated metal-housed mass flow control (MFC) systems for three different gases. The MFCs allow software-controlled gas switching and purge gas rates as well as recording of the flow rates in the software.

## Various furnace systems

The DSC 404 **F1 Pegasus**<sup>®</sup> can be equipped with a wide range of different furnaces accommodating different temperature and application ranges. A double furnace hoist allows the simultaneous installation of two different furnaces for improved sample throughput or low- and high-temperature tests with the same instrument. The furnaces can be easily changed by the operator. Therefore, the system is adaptable to any future application range.



## Furnaces:

Furnace type	Temperature range	Cooling system
Steel furnace	-150 ... 1000°C	liquid nitrogen
Platinum furnace	RT ... 1500°C	forced air
Silicon carbide furnace	RT ... 1550°C	forced air
Rhodium furnace	RT ... 1650°C	forced air
Graphite furnace	RT ... 2000°C	tap water

### Various sensors

The DSC 404 **F1 Pegasus**® is generally used to obtain accurate specific heat measurements. However, the system allows for simple DTA measurements or conventional DSC tests as well. DTA sensors can be used for applications such as routine tests on aggressive sample substances. Various thermocouple types allow optimum sensitivity and time constants in all temperature ranges.

### Unique sensor adjustment system

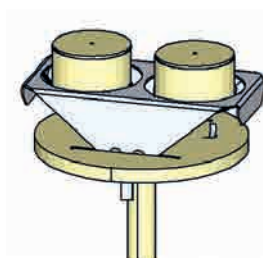
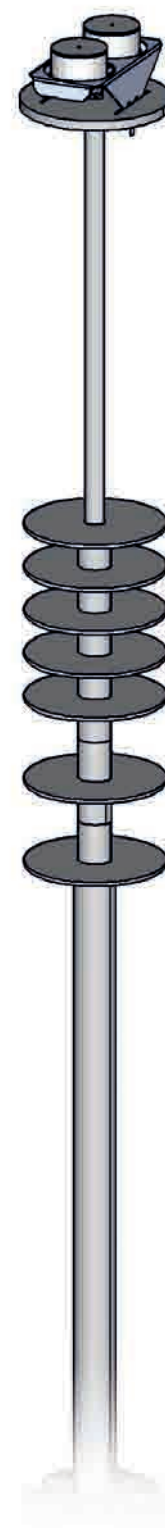
For optimizing the baseline, a micrometer adjustment system is integrated into the measurement part. This adjustment system allows placement of the sensor at the optimum central position in the furnace. This guarantees a stable and reproducible baseline without any major adjustment efforts.

### Automatic sample changer

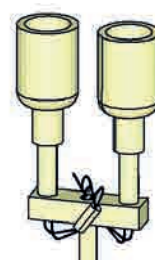
An automatic sample changer for up to 20 samples is optionally available. The sample changer guarantees optimum crucible placement and effective use of the instrument. Pre-programming allows measurements to be carried out during the night or over a weekend. The software can automatically carry out analyses using predefined macros.

### BeFlat®, DSC correction and TM-DSC

Innovative software features such as *BeFlat*® and DSC correction allow a fully automatic baseline correction as well as correction for system time constants. All routines are fully software-based. This can be optimized for your specific measurement conditions. Furthermore, temperature-modulated DSC (TM-DSC) tests can be carried out (not available in North America and Japan).



DSC-c<sub>p</sub>



DTA

### Sensors:

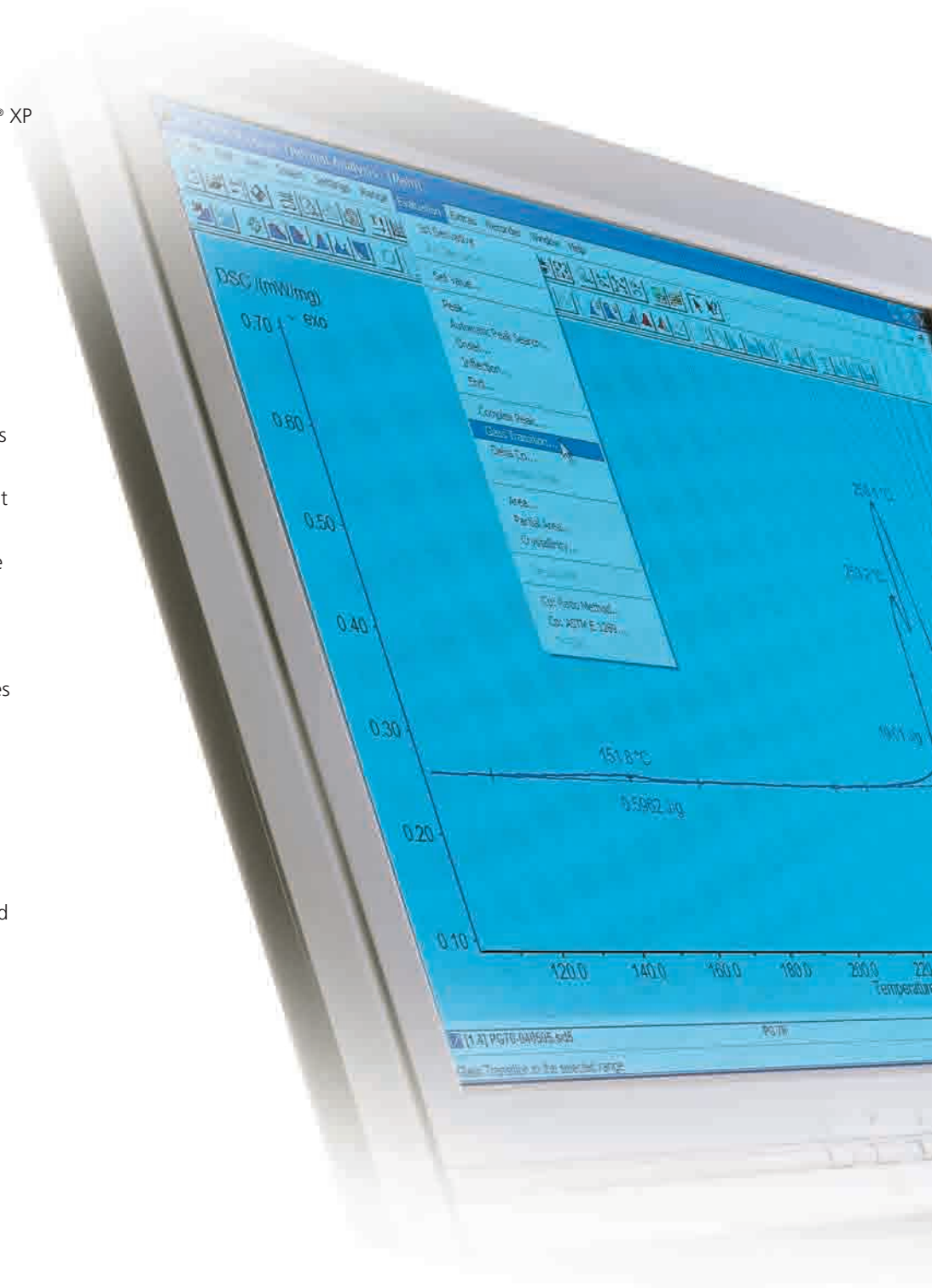
Sensor thermocouple	Temperature range	Sensor types	Atmospheres
Type E	-150 ... 700°C	DTA, DSC-c <sub>p</sub>	inert, oxid. (to 500°C), vac.
Type K	-150 ... 800°C	DTA, DSC-c <sub>p</sub>	inert, oxid. (to 600°C), red., vac.
Type S	RT ... 1650°C	DTA, DSC, DSC-c <sub>p</sub>	inert, oxid., red., vac.
Type B	150 ... 1750°C	DTA, DSC	inert, oxid., red., vac.
Type W	RT ... 2000°C	DTA	inert, red., vac.

# DSC 404 **F1 Pegasus**<sup>®</sup> – **Proteus**<sup>®</sup> Software

The DSC 404 **F1 Pegasus**<sup>®</sup> runs under a 32-bit Windows<sup>®</sup> software package which includes everything you need to carry out a measurement and evaluate the resulting data. Through the combination of easy-to-understand menus and automated routines, a tool has been created that is extremely user friendly and, at the same time, allows sophisticated analysis.

## General Software Features:

- Windows<sup>®</sup> software: for Windows<sup>®</sup> XP and Vista<sup>®</sup> (Enterprise, Business) operating systems
- Multi-tasking: simultaneous measurement and evaluation
- Multi-moduling: operation of different instruments with one computer
- Combined analysis: comparison and/or evaluation of DSC, TGA, DIL, TMA and DMA measurements in one plot
- Labeling: input and free placement of text elements
- Calculation of 1<sup>st</sup> and 2<sup>nd</sup> derivative
- Selectable scaling
- Graphic and data export
- Selectable colors and line types
- Storage and restoration of analyses
- Macro recorder (optional)
- Context-sensitive help system
- Temperature calibration
- Compatible with advanced software packages (Peak Separation, *Thermokinetics*)
- Software produced by ISO-certified company

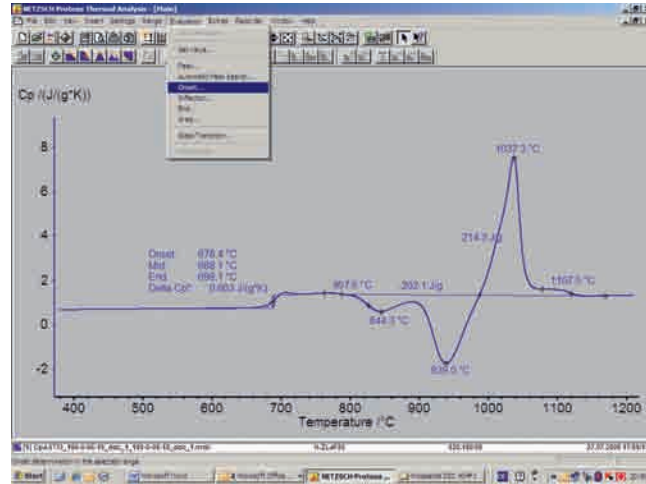


Certified ISO 9001 by



## DSC Features:

- Determination of onset, peak, inflection and end temperatures
- Automatic peak search
- Transformation enthalpies: analysis of peak areas (enthalpies) with selectable baseline and partial peak area analysis
- Comprehensive glass transition analysis
- Automatic baseline correction
- Degree of crystallinity
- O.I.T. (oxidative induction time) evaluation
- Specific heat determination
- *BeFlat*<sup>®</sup> for automatic baseline correction
- DSC correction: evaluation of exo- and endo-thermal effects under consideration of system time constants and thermal resistance values



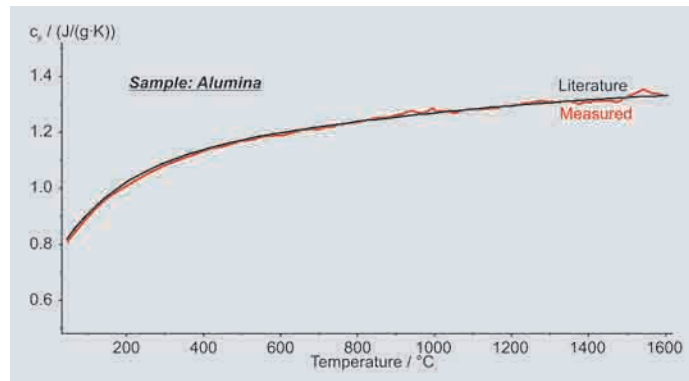
## Advanced Software (options)

- TM-DSC module software extension for temperature-modulated DSC tests (This feature is not available in North America and Japan.)
- Peak Separation Software: allows accurate separation and evaluation of overlapping transitions
- NETZSCH *Thermokinetics*: allows advanced characterization of reactions and kinetic parameters on the basis of multiple-step kinetic analysis on up to 16 curves, also provides predictions of the process
- Purity determination
- *Proteus<sup>®</sup>Pharm<sup>™</sup>* allows measurements and evaluations according to 21 CFR Part 11

# DSC 404 **F1** Pegasus® – Applications

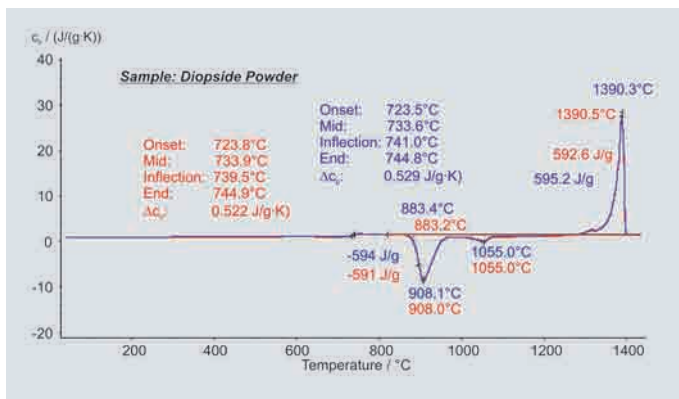
## Specific heat accuracy of alumina (Al<sub>2</sub>O<sub>3</sub>)

Presented in the figure is a specific heat test result for a polycrystalline alumina sample between room temperature and 1600°C. Additionally shown are literature values for pure alumina. It can clearly be seen that there are no significant differences between the literature values and the test results. The maximum deviations are in the range of 2%, which demonstrates the outstanding performance of the DSC 404 **F1** Pegasus®.



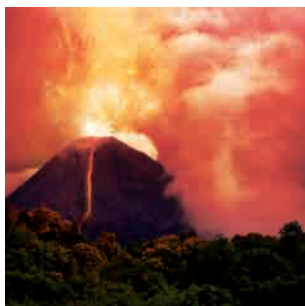
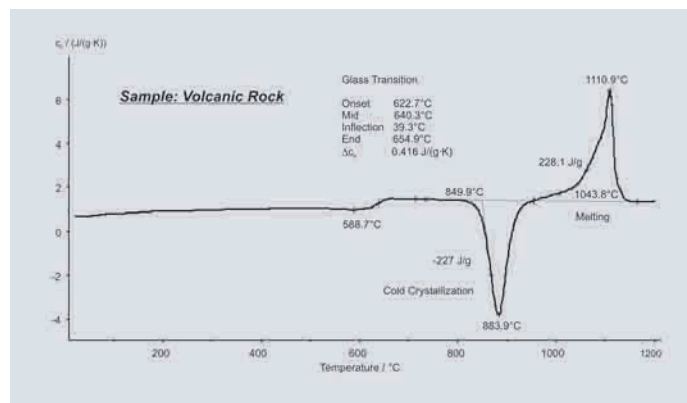
## Reproducibility of a diopside glass powder

Presented here are two measurement results for diopside glass powder. The glass transition occurred between 723°C and 745°C. Crystallization was measured at 883°C (onset temperature). Melting took place at 1390°C (main peak temperature). The characteristic temperatures and the corresponding enthalpy changes are in good agreement between the two different samples. Furthermore, the specific heat itself doesn't show relevant differences. This indicates the excellent stability and reproducibility of the DSC 404 **F1** Pegasus® system.



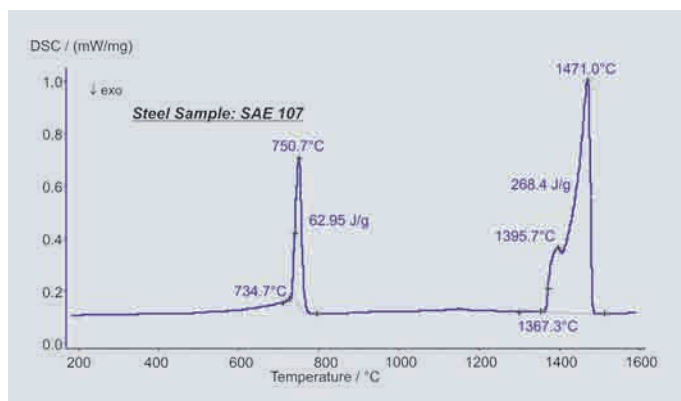
## Specific heat of volcanic rock

Natural materials such as rocks are difficult to analyze with regard to their chemical composition. Volcanic rocks are generally solidified from molten magma and their main contents are different oxides. This example shows a DSC measurement result for such a material. It can clearly be seen that it solidified almost completely amorphous. The glass transition was determined between 623°C and 655°C. Cold crystallization and melting were detected at 884°C and 1111°C (peak temperature). The heat released during crystallization was comparable to the heat of fusion, indicating the highly amorphous nature of this mixture.



## Phase transitions in an SAE 107 steel material

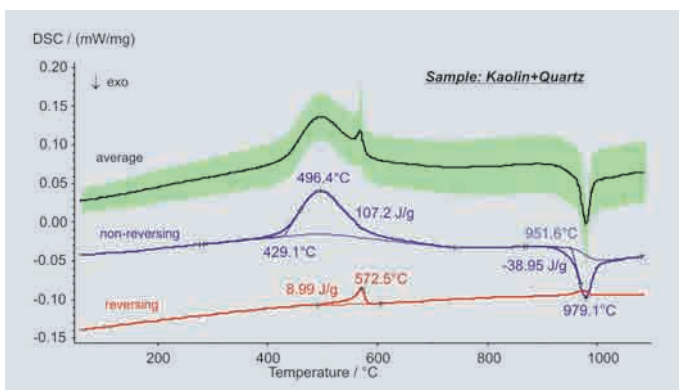
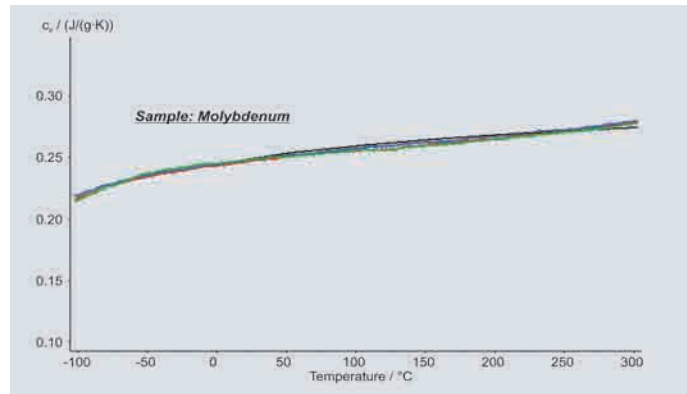
Presented here are the transformation energetics of a steel (SAE 107). At 751°C, two phase transitions overlap each other. The increase in the heat flow rate up to 735°C is due to the Curie transition (change in the magnetic properties). A change in the crystal structure (bcc to fcc structure) is causing the major peak. The structural change is connected with an enthalpy change of 63 J/g. Melting was seen at 1367°C and occurred in two steps (peaks at 1395°C and 1471°C). The heat of fusion was 268 J/g.



# DSC 404 **F1** Pegasus® – Applications

## Specific heat of molybdenum

The specific heat of molybdenum was measured three times between -100°C and 300°C using the new low-temperature furnace of the DSC 404 **F1** Pegasus®. The three results (colored lines) show only slight scattering in the range of 2%. Additionally shown are literature values for pure molybdenum (black line) between room temperature and 300°C. The differences between the measurement results and the literature values are generally less than 2%. This proves the excellent specific heat performance of the system in the low-temperature range.



## Characterization of a kaolin-quartz mixture

Temperature-modulated DSC (TM-DSC) is a tool generally employed for low-temperature applications on polymers. The DSC 404 **F1** Pegasus® is the first DSC capable of doing temperature modulation at high temperatures. Presented here are measurement results for a kaolin-quartz mixture. The irreversible dehydration and phase changes of the kaolin were detected in the non-reversing part of the DSC result. The quartz transition at 573°C was detected in the reversing part of the DSC curve. (This feature is not available in North America and Japan.)

# DSC 404 **F1** Pegasus® – Servicing Our Customers' Special Needs

## Accessories

A wide range of crucibles (aluminum, silver, gold, copper, platinum, alumina, zirconia, graphite, stainless steel, etc.) is available for nearly all possible applications and materials.

For working in critical atmospheres, a "CO version" of the DSC 404 **F1** Pegasus® can be supplied. This version is optimized for measurements under corrosive or reducing atmospheres. Gas flow control systems are prepared in a separate box and special sensors with protected thermocouple wires are available.

For measurements on difficult samples or radioactive substances, the DSC 404 **F1** Pegasus® can be prepared for installation in a glove box or hot cell. Electronics are removed from the measurement part and all cables and fittings are prepared for connection to an existing feedthrough.

If you have any other special application or test condition, ask us! Our engineers are prepared to develop special versions of instrumentation or software with your requirements in mind.

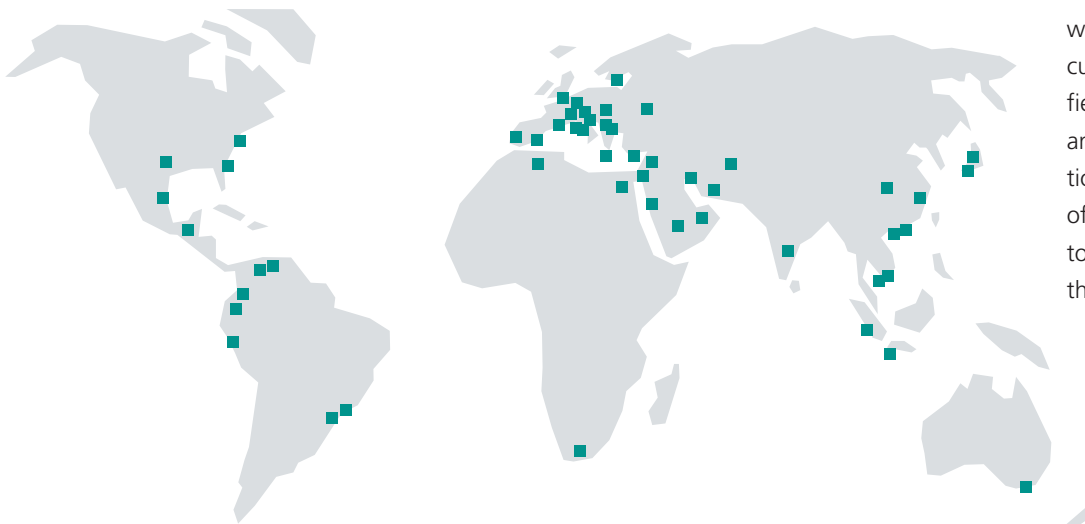


## Global Customer Support & Service Network

State-of-the-art technology combined with optimal customer support are NETZSCH trademarks. Our training department provides a complete range of programs tailored to the needs of our customers in research, education and industry.

A wide range of different seminars, users' meetings and individual training programs is available to assist you in achieving optimum performance and benefit from your thermal analysis system.

NETZSCH is the fastest growing company in the field of thermal analysis and thermophysical properties testing in the world. This can be attributed not only to our superior technology and quality, but also to our unmatched pre- and after-sales service network. NETZSCH-certified staff at 45 service centers across the world provide fast and reliable customer support including qualified installation, calibration services, and maintenance contracts. In addition, our applications laboratories offer contract testing and support to address the most specific of thermal analyses.



# Pegasus®

## Advantages in DSC Testing

The new DSC 404 **F1 Pegasus**® is the ideal tool for day-to-day work in your laboratory. The system is generally employed for:

- Top-level research
- Material development
- Quality assurance
- Failure analysis

The DSC 404 **F1 Pegasus**® is part of the NETZSCH high-temperature series of instruments. Together with the DIL 402 PC/C (dilatometer), the TMA 202/402 (thermomechanical analyzer), the DMA 242 C (dynamic mechanical analyzer), the STA 449 *Jupiter*® (simultaneous thermal analyzer), the DEA 230/231 series (dielectric analyzers) for cure monitoring, and other DSC and TGA systems, a full-scale thermal analysis of your materials and parts can be carried out.

The key features of the DSC 404 **F1 Pegasus**® are:

- Maximum flexibility
- Top quality
- Optimum performance
- Wide range of accessories
- Low cost of ownership

NETZSCH offers a full range of low and high temperature thermal analysis instruments for temperatures between -260 and 2800°C, including all conventional thermal analysis systems.

For thermophysical properties testing (measurement of the thermal diffusivity/conductivity), NETZSCH offers a broad range of heat flow meters (HFM), guarded hot-plate systems (GHP), flash devices (LFA) and other thermal conductivity testers (TCT).

Technical specifications subject to change

## Leading Thermal Analysis .

# NETZSCH

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