

Leading Thermal Analysis ■

**Determination of the Aging Resistance of Polymers with
Standardized Tests for Thermo-Oxidative Aging,
OIT Determination with Differential Scanning Calorimeter (DSC)**

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Determination of the Aging Resistance of Polymers with Standardized Tests for Thermo-Oxidative Aging,
OIT Determination with Differential Scanning Calorimeter (DSC)

1. External factors influencing aging of polymers

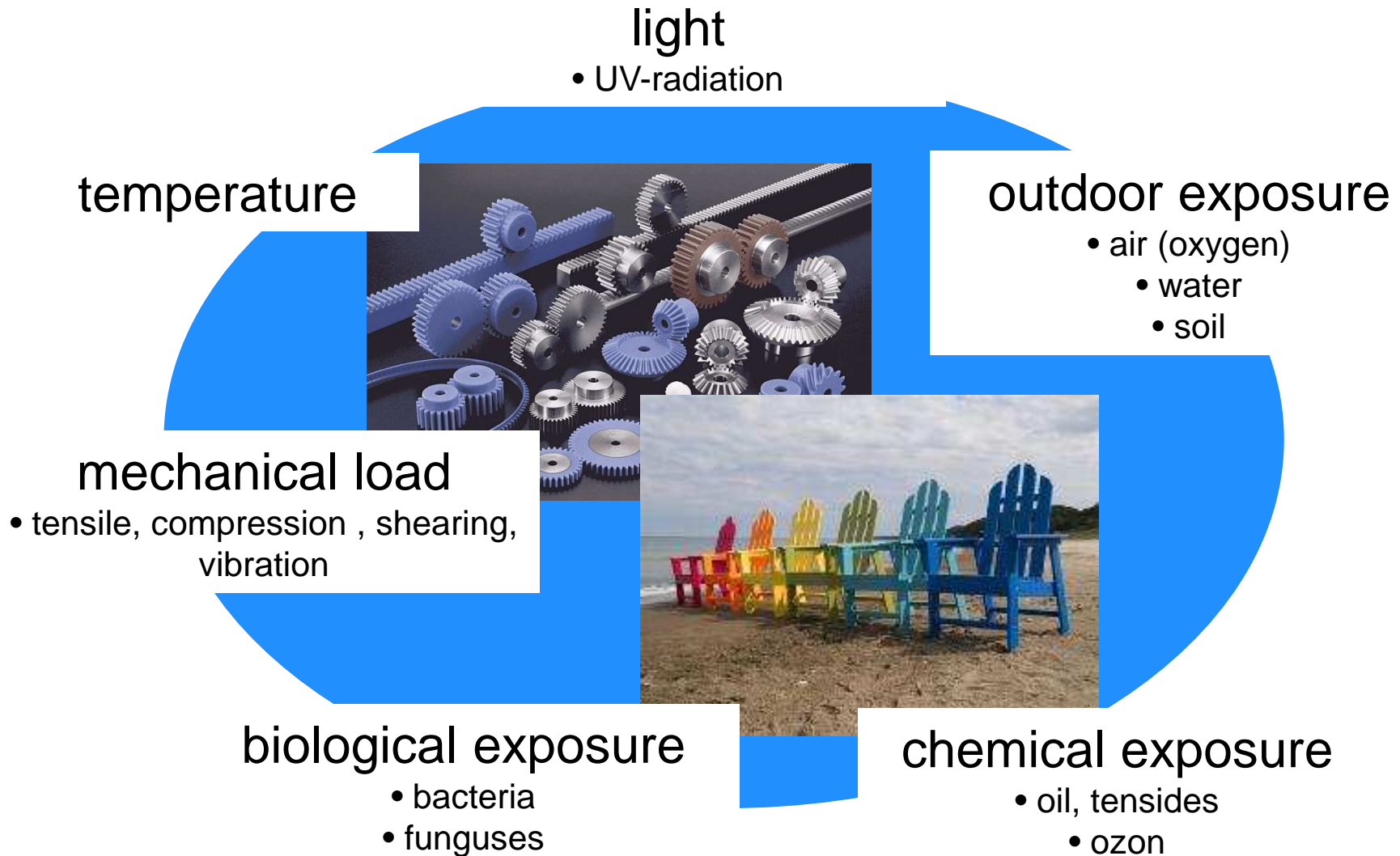
2. Possibilities to reduce aging

3. Oxidative-InductionTime (OIT)

- Standards
- OIT determination with DSC

3. Failure analysis using OIT measurements

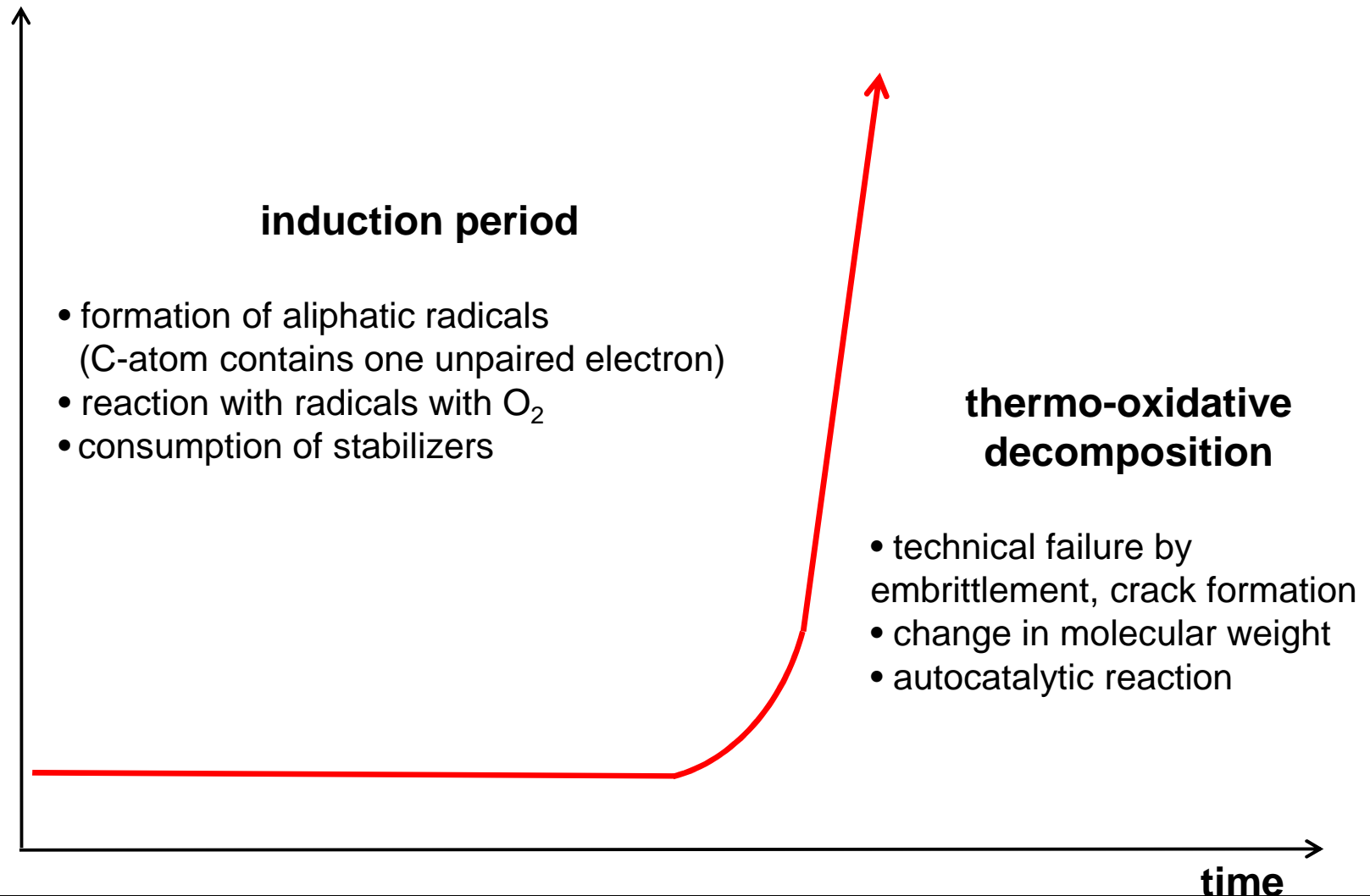
4. Summary



Most important stress types influencing long-term use of polymers

- **Oxidation**
thermo-oxidative (T, O₂) and photo-oxidative aging (hf, T, O₂)
 - **Influence of chemicals and simultaneous mechanical stress**
 - **Biological aging**
-

change in material properties



Appropriate Stabilizers protect polymers against damage by:

- **oxygen (air)**(O_2)
- **heat** (T)
- **light** (hf)
- **shearing** (τ)
- **metal ions** (M^{n+})

Stabilizers slow down the aging process and extend the induction period

- **Antioxidants**

(primary antioxidants) protect the polymer during production and molding as well as from influences caused by heat and oxygen.

- **Co-stabilizer**

(secondary antioxidants) support the efficiency of the primary antioxidants

- **light stabilizer**

increase the resistance against light (UV)

- **metal deactivator**

minimize the influence of metal contact: Cu cable

ASTM D 3895

Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry

ASTM E 2009

Standard Test Method for Reaction Induction Time by Thermal Analysis

ASTM D 4565

Standard Test Method for physical and environmental performance properties of insulations and jackets for telecommunications wire and cable

ASTM D 525

Standard Test Method for Oxidation Stability of Aviation Fuels

ASTM E 487-79

Standard Test Method for constant temperature stability of chemical materials

ISO 11357-6

Determination of oxidation induction time by DSC

EN 728

Plastic piping and ducting systems – Polyolefin pipes and fittings – Determination of oxidation induction time

Determination of Oxidative Induction Time OIT with Differential Scanning Calorimeter - DSC

NETZSCH

Pre-condition: polyolefin contains antioxidants and other stabilizers

measurant: the time (induction period) to prevent oxidation of the material



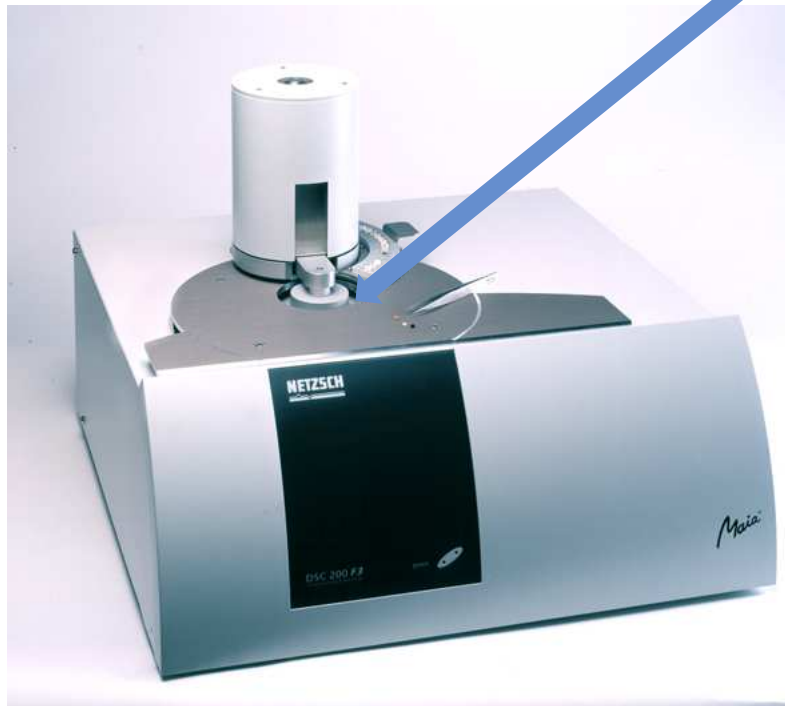
DSC 214 Polyma
with automatic sample changer
Temperature range: -170°C....600°C



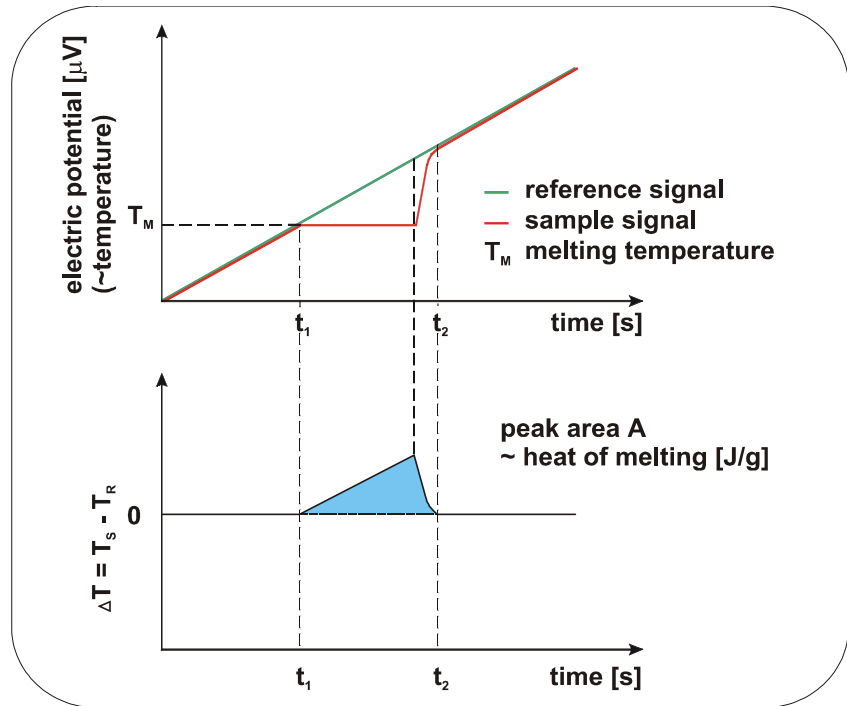
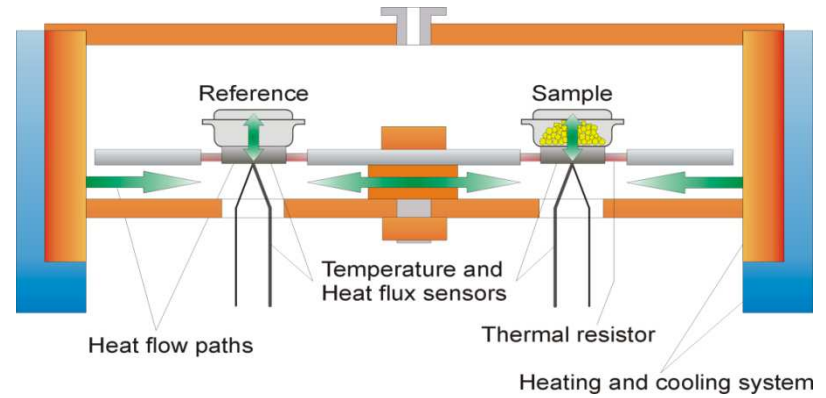
DSC 204 F1 Phoenix
with automatic sample changer
Temperature range: -180°C....700°C

DSC (Differential Scanning Calorimetry)

measuring cell

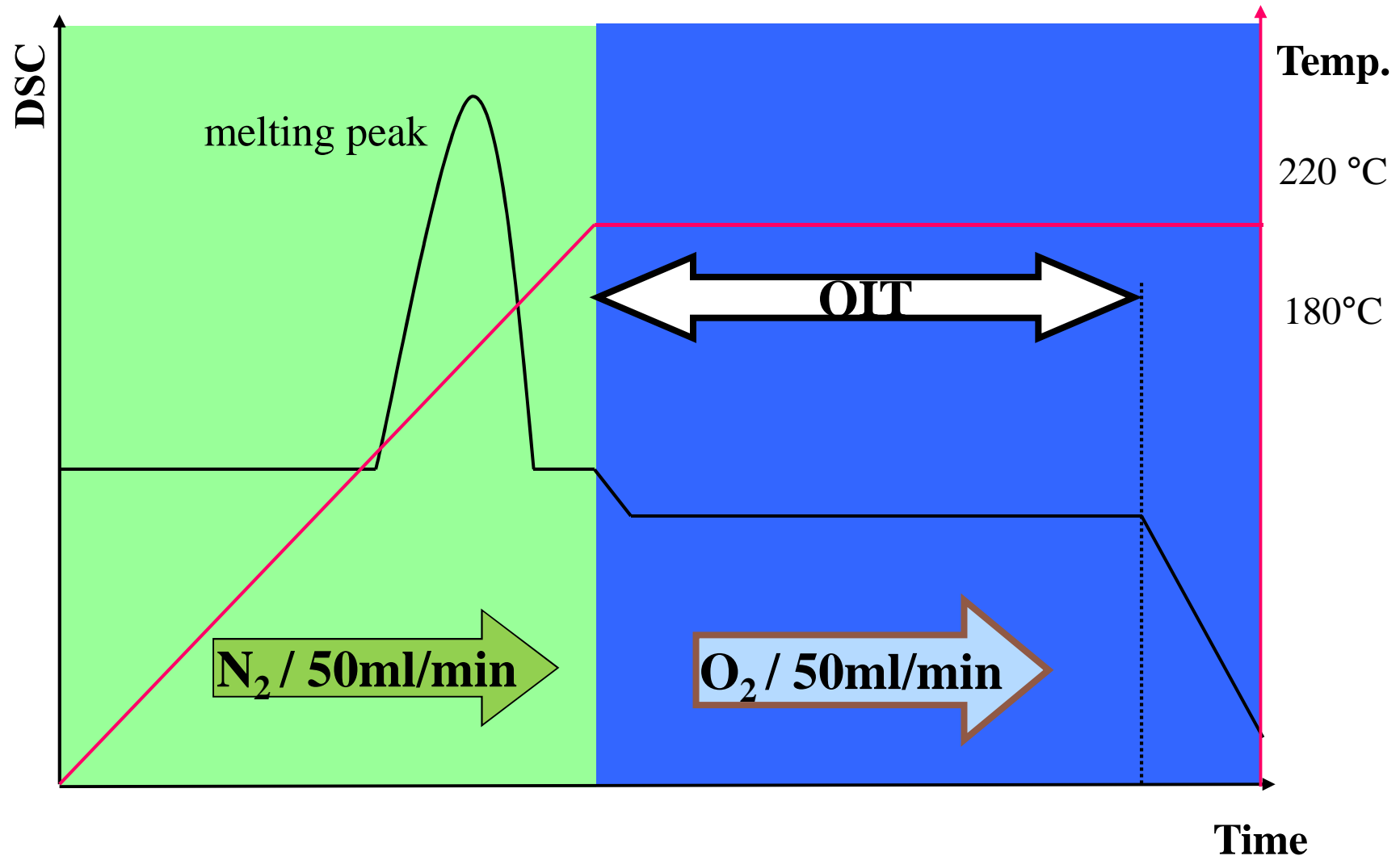


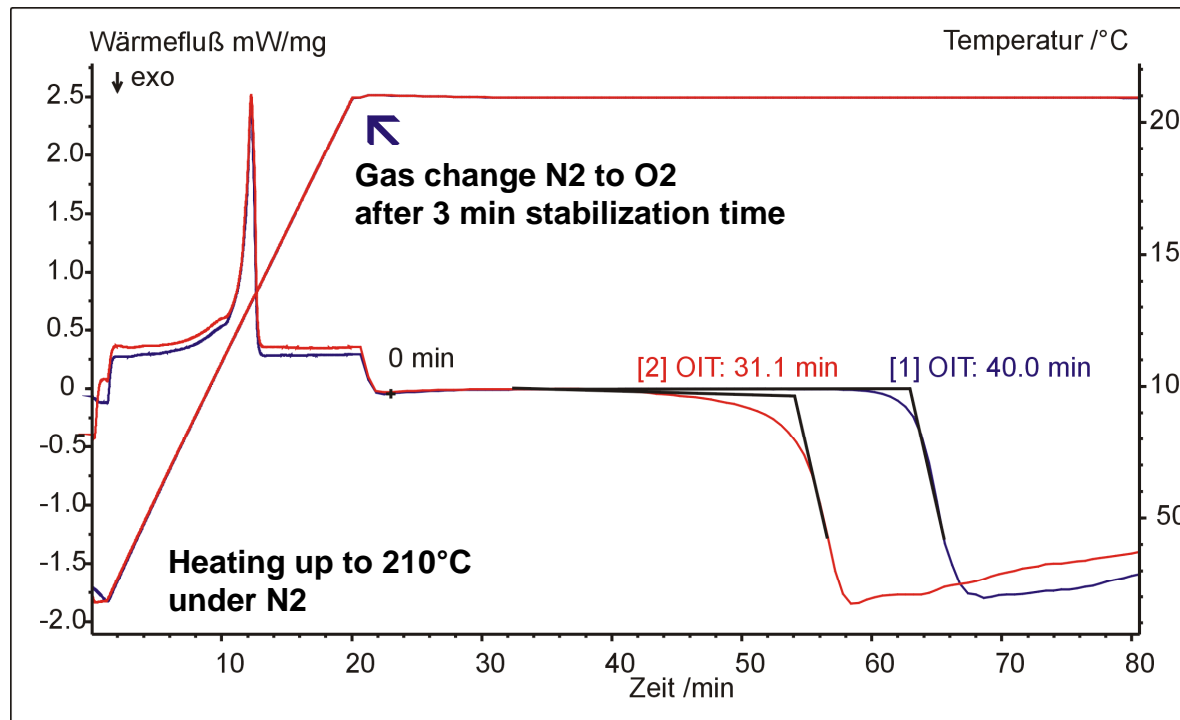
signal generation



OIT – Measurement

Isothermal Measurement According to DIN-EN 728





Sample: (1) PE tube
smooth surface
(2) PE tube
rough surface

Mass: (1) 7.2 mg
(2) 7.67 mg

Crucible: Al, pierced lid

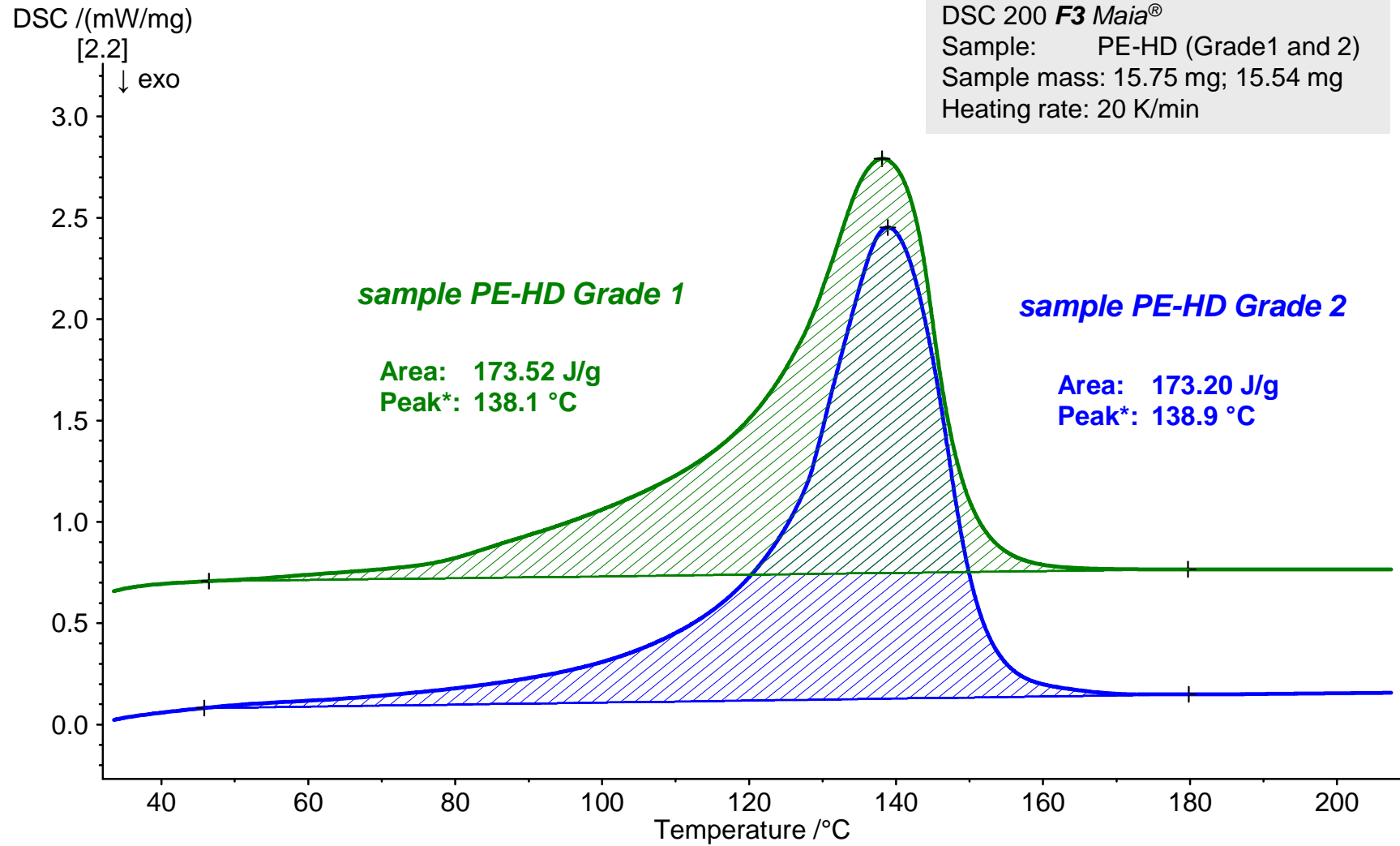
Heat. rate: 20 K/min
isothermal

Atmosphere: N2/O2

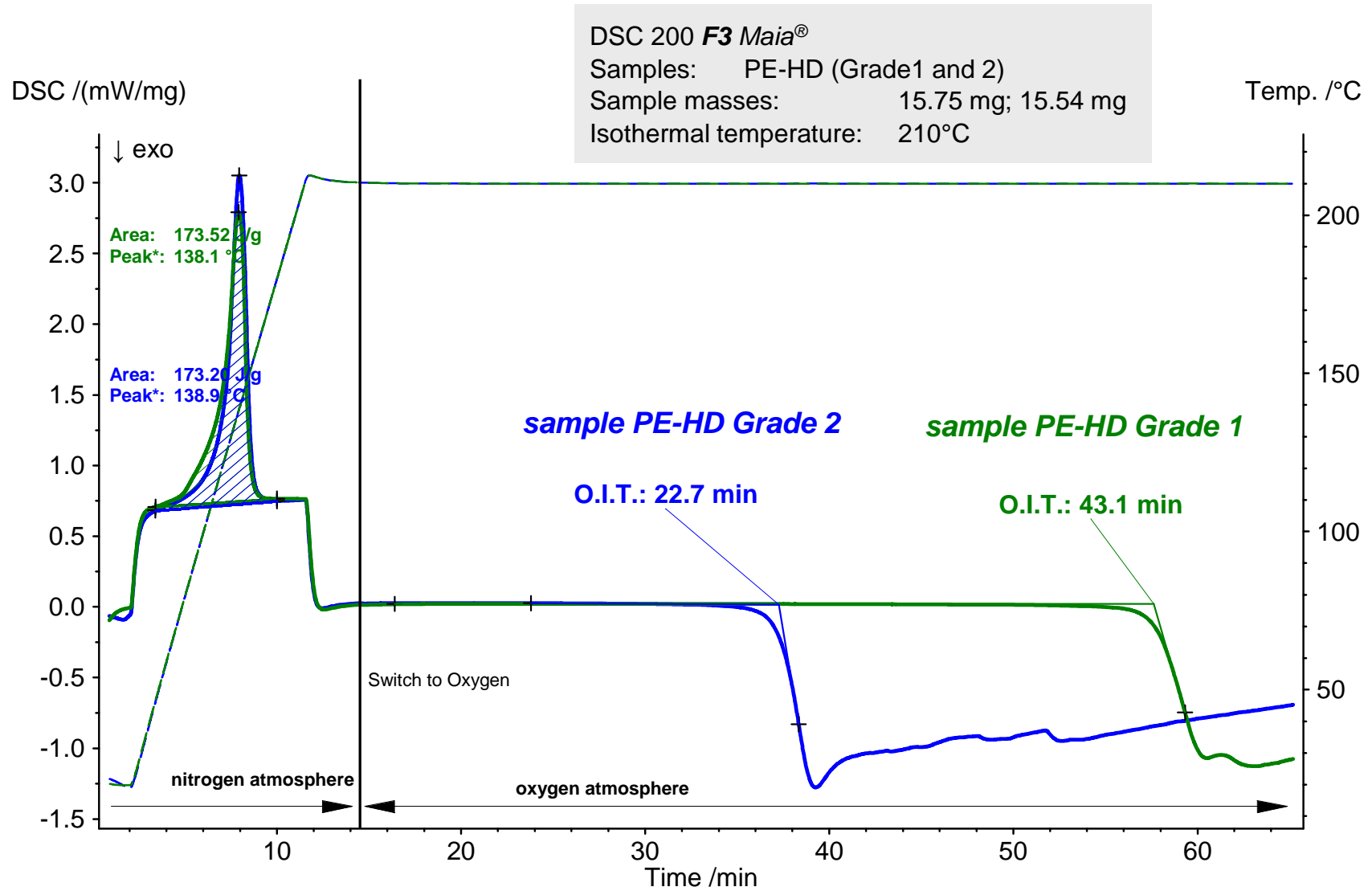


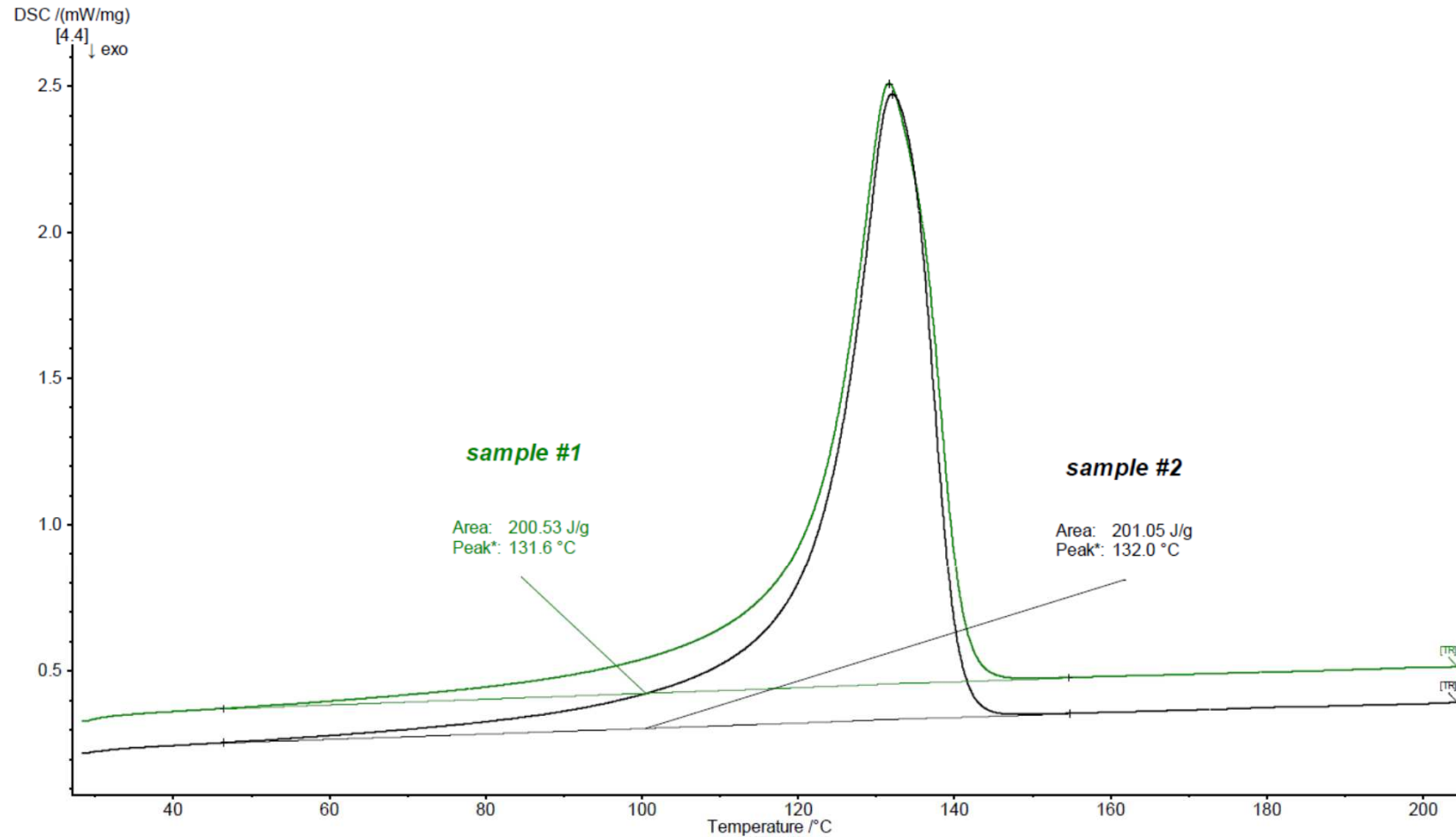
O.I.T. measurement according to DIN EN 728, ISO 11356-6, ASTM D 3895 (normally with open Al-or Cu-pans, Cu-pans for cables). Isothermal temperature depends on standard.

Melting of PE-HD (Two Grades)

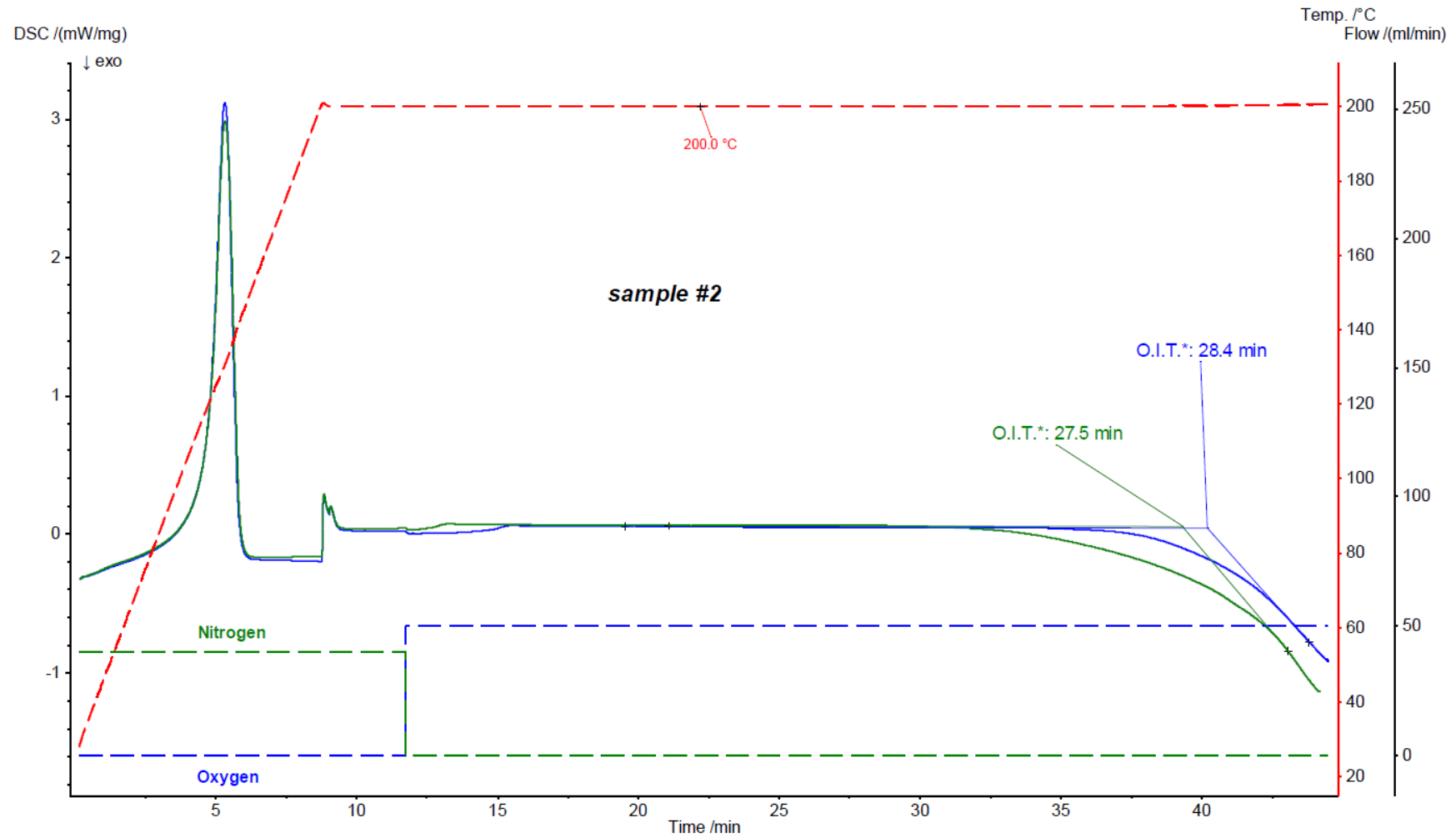


OIT Measurement on PE-HD (Two Grades)

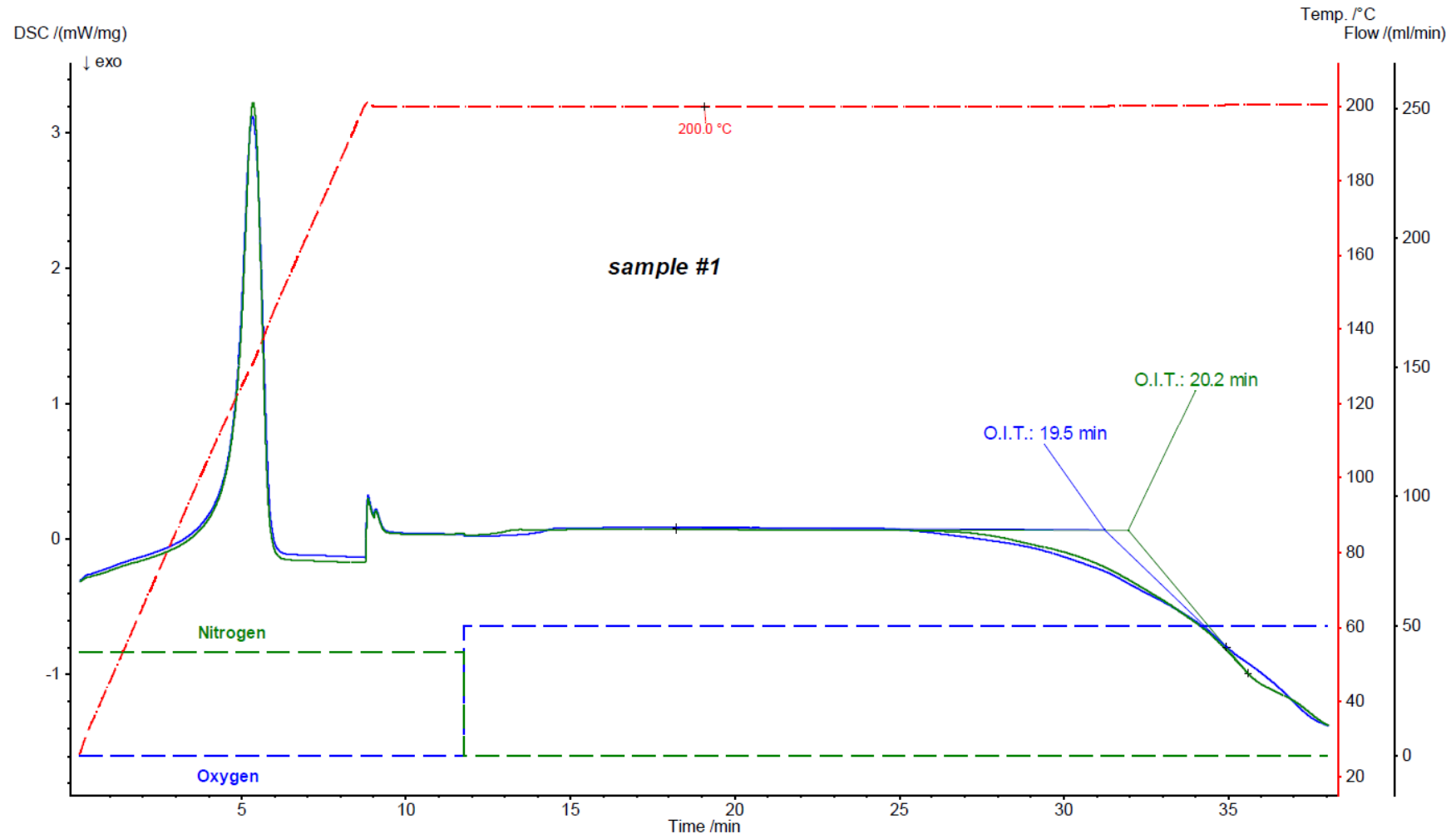




■ No difference during melting

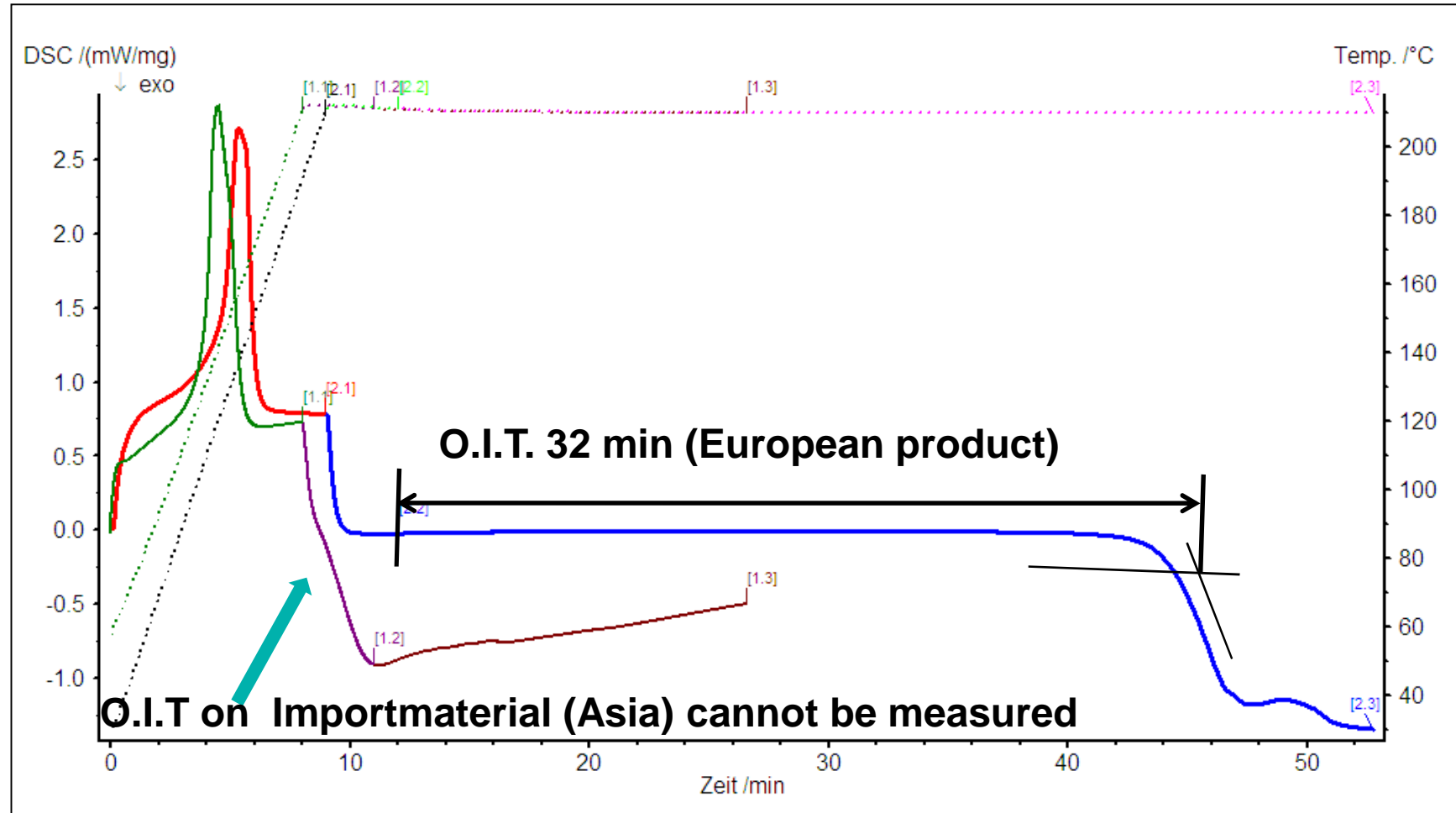


■ OIT reproducibility for sample 2

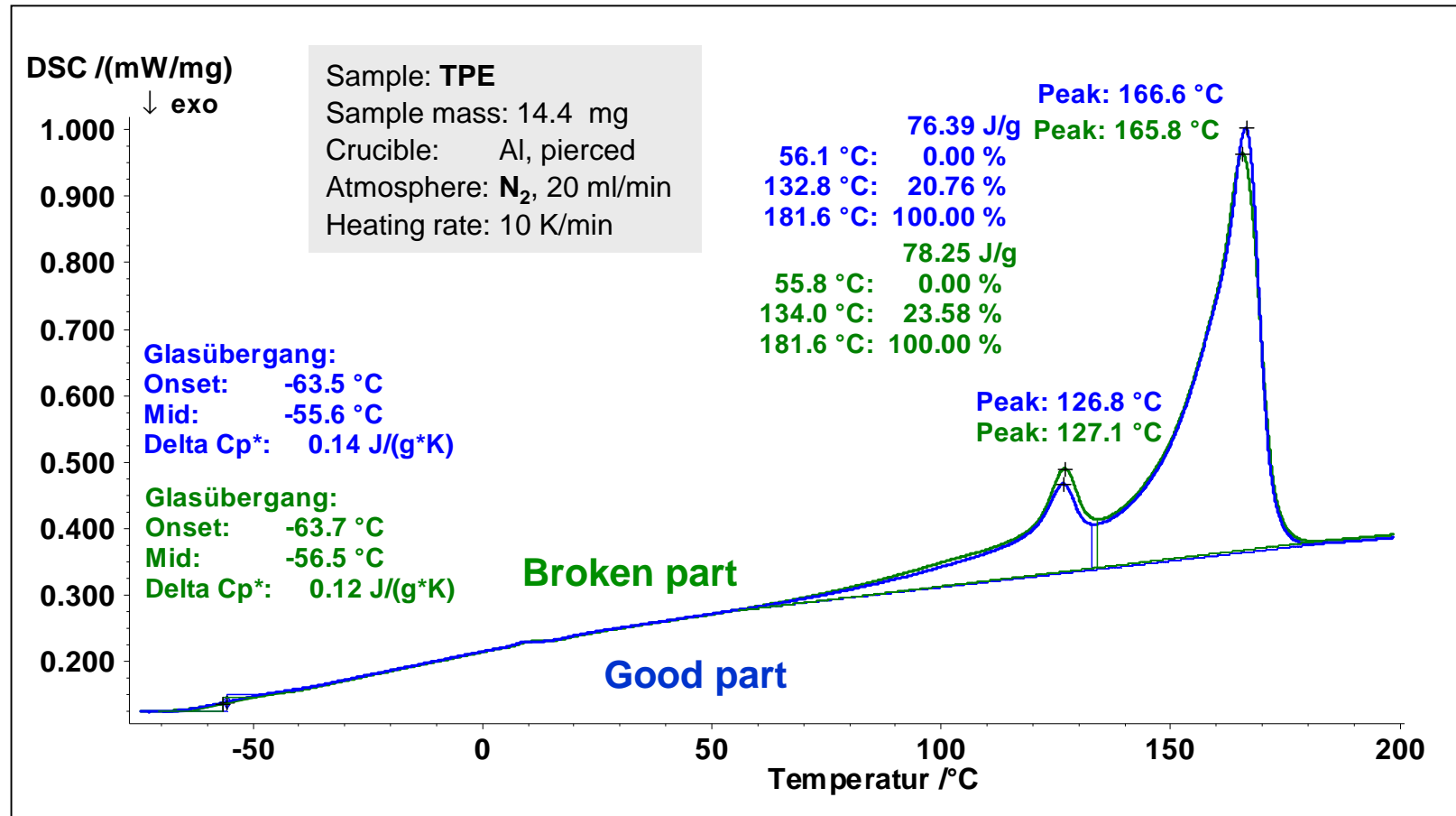


- OIT reproducibility for sample 1 (about 8 min lower than sample 2)
- Sample 1 has a lower stability against oxidation.

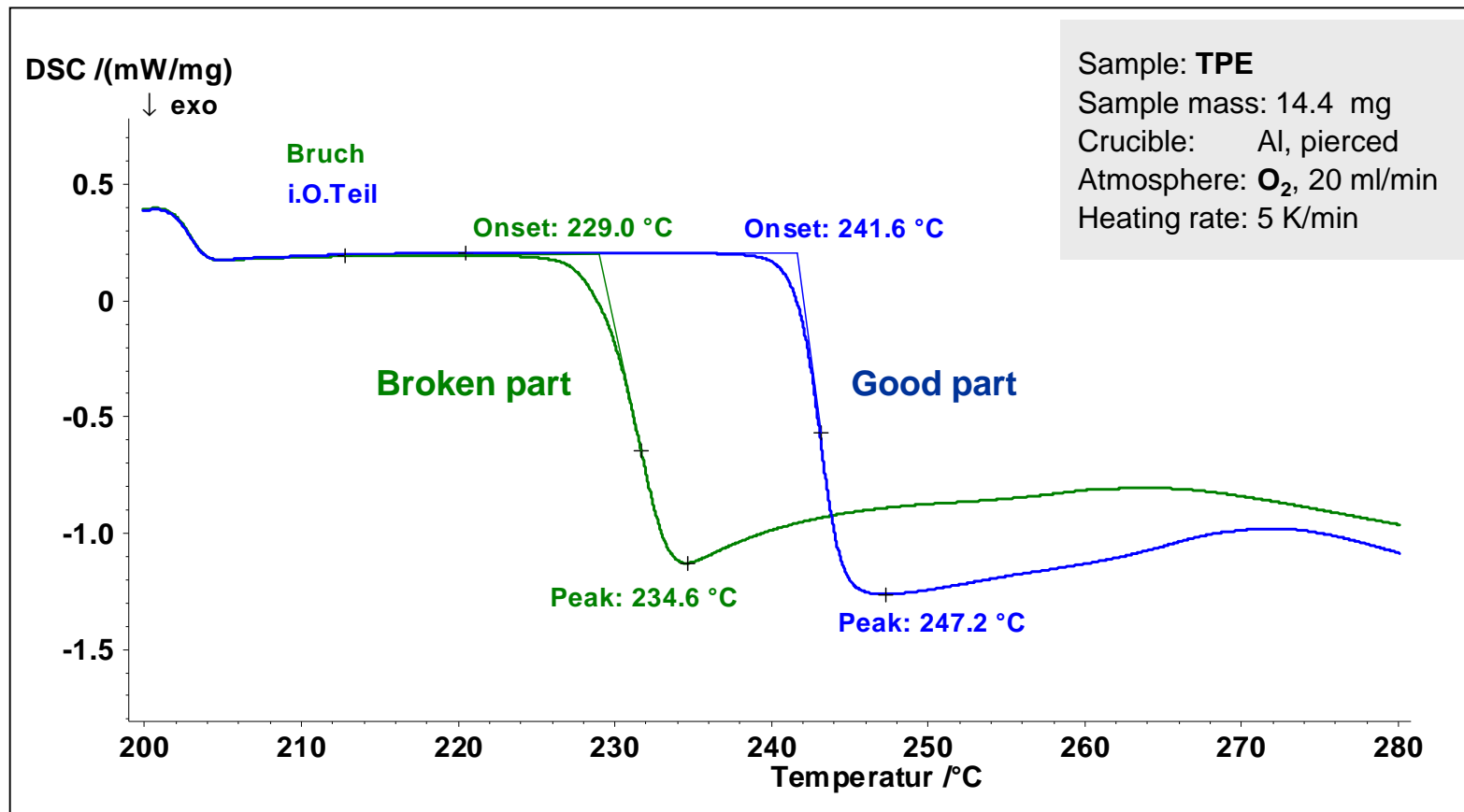
Failure Analysis with OIT Measurement - PE Parts Used in a Fish Plant in Norway-



As soon as the change from nitrogen to oxygen happens oxidation occurs.



1. No significant differences between **good** and **broken** part with normal heating in nitrogen.



2. Change to oxygen: excellent separation of material properties by dynamic OIT

O.I.T. determination is a very easy and relatively fast method for quality control and failure analysis.

The method (isotherm) corresponds to international standards applicable for polyolefin. With modifications it can also be used for other polymer types

Long-term predictions over several years based on OIT only, however, should be rated as critical.
