



SUSTAINABLE ADDITIVES TO INCREASE THERMAL STABILITY OF DRY LUBRICANTS AND BEST PRACTICE OF USE

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Wire & Cable, Milan - 16/10/2023

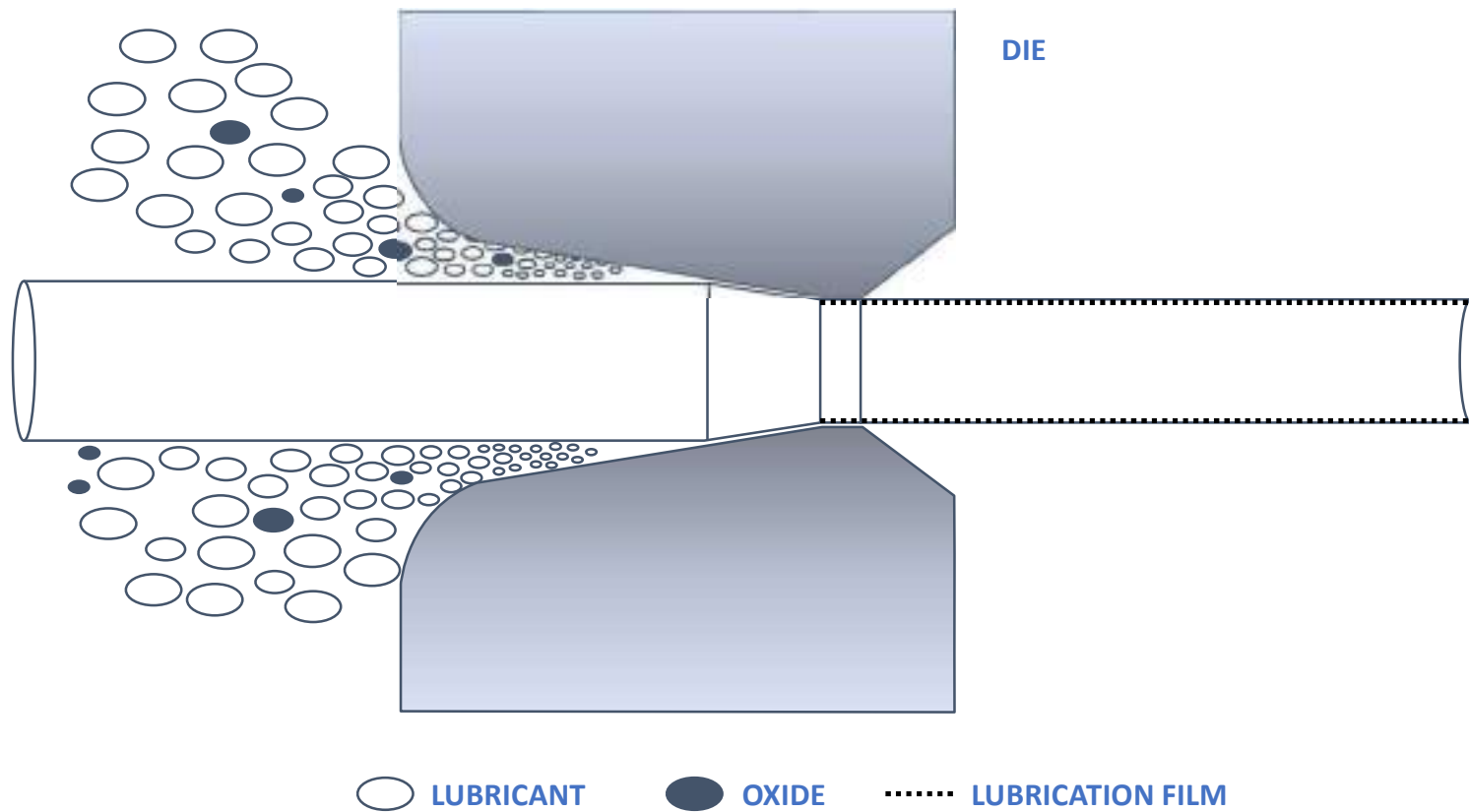


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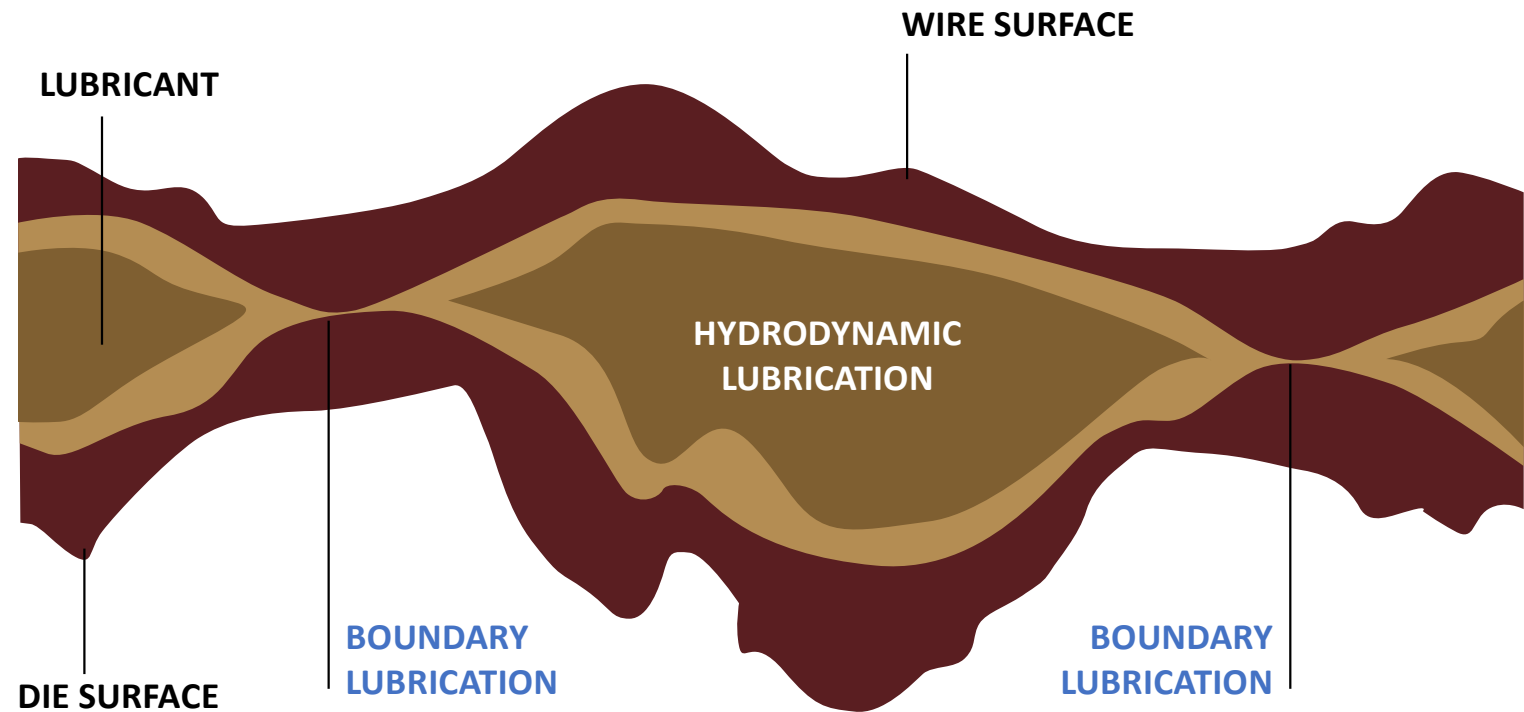


THEORY ABOUT WIRE DRAWING

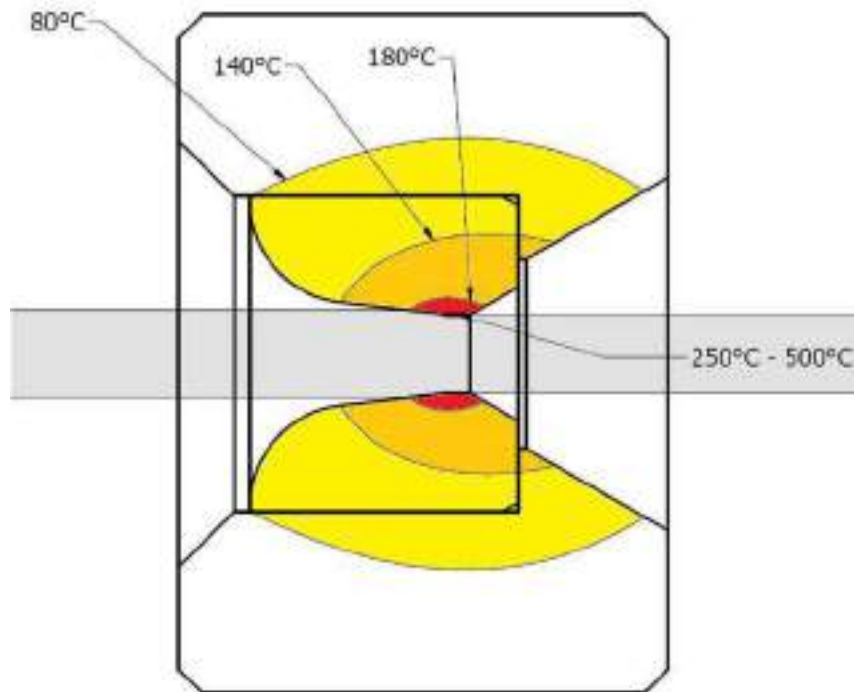


THEORY ABOUT WIRE DRAWING

- Lubricant reduces friction between the surface of the wire and the die
- Hydrodynamic lubrication: thick layer, total separation between wire and die
- Boundary lubrication: thin layer between wire and die



THEORY ABOUT WIRE DRAWING



- Cold-forming process
- Lubricant is essential to reduce friction and wear in the contact zone, reducing temperature
- Too high temperatures can modify and embrittle the microstructure of the steel
- Lubricants must have high thermal resistance

THERMAL RESISTANCE OF SODIUM SOAPS: SAMPLE PREPARATION

Sodium Soap
+
3 wt.% Flame Retardant

Starting fatty acid:
Stearin

70% C18, Stearic Acid

30% C16, Palmitic Acid

SAMPLE

Sodium Stearate (Stearin, C16 + C18)

Sodium Palmitate (C16)

Sodium Stearate (Pure Stearic Acid, C18)

Borax Pentahydrate

Sodium Metasilicate Pentahydrate

Melamine

Aluminium Hydroxide

Sodium Phosphate

Aromatic Phosphate Esther

Sodium Nitrate

Sodium Organic Salt

Sodium Organic Salt + Sodium Metasilicate
Pentahydrate

THERMAL RESISTANCE OF SODIUM SOAPS: DIFFERENTIAL SCANNING CALORIMETRY (DSC)

- Measure variation of heat absorbed by the material during a variation of its state
- Phase transition, melting point and oxidation
- Observed phenomena can be endothermic or exothermic

INHERT ATMOSPHERE

Atmosphere: Nitrogen

Ramp from 30°C to 400°C: 5°C/min

Pan: Aluminium

Sample weight: 10-20 mg

OXIDATIVE ATMOSPHERE

Atmosphere: Air

Ramp from 30°C to 400°C: 5°C/min

Pan: Aluminium

Sample weight: 10-20 mg



PerkinElmer DSC 8000 coupled with Intracooler 2

THERMAL RESISTANCE OF SODIUM SOAPS: DIFFERENTIAL SCANNING CALORIMETRY (DSC)

NITROGEN

- Inert environment
- Cleaner graph
- Less similar to real conditions
- Better to study crystalline phases

AIR

- Reactive environment
- Oxidative reactions (Like burning)
- 'Noisy' graph
- Replicate real conditions

Energy involved in these transitions is the enthalpy:

$$\Delta H = K A$$

K is the calorimetric constant and A is the area under the curve

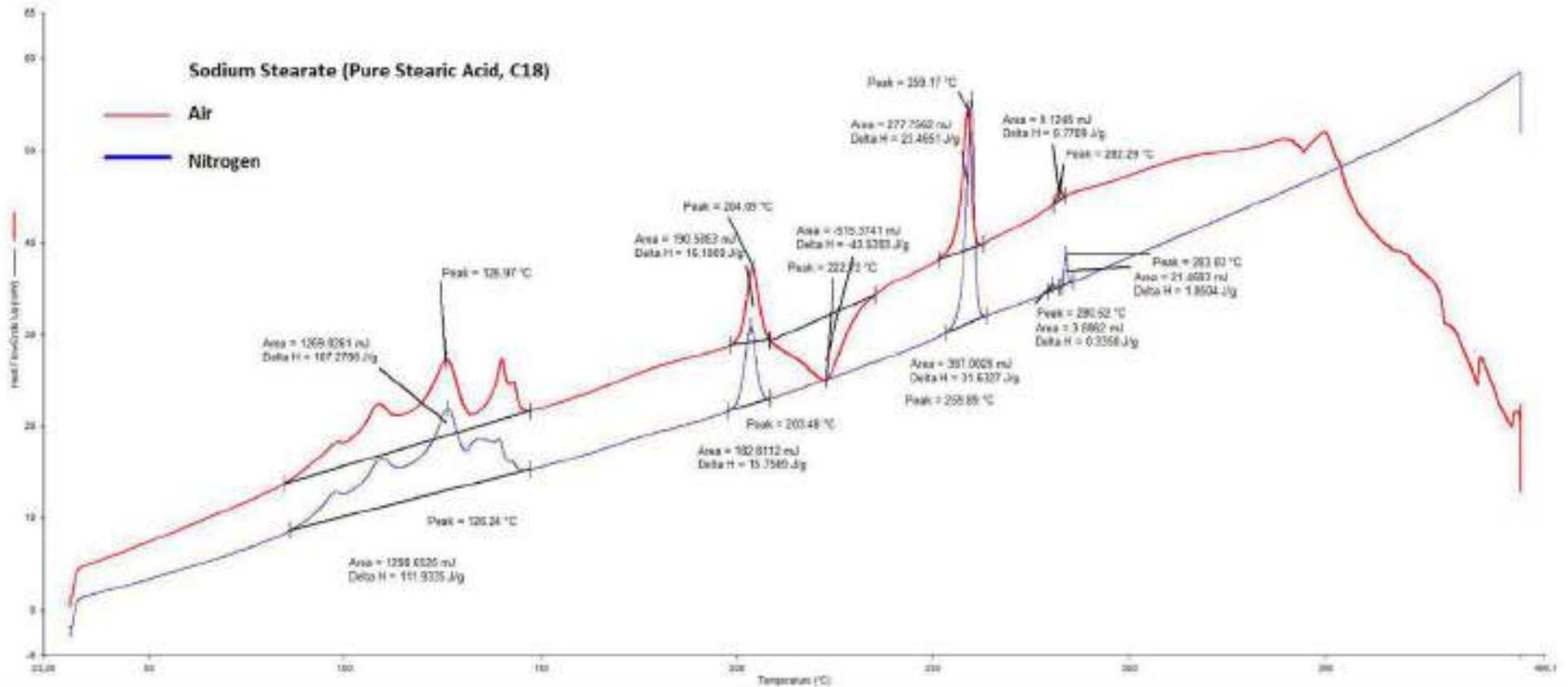
THERMAL RESISTANCE OF SODIUM SOAPS: DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Phases of pure sodium soap:

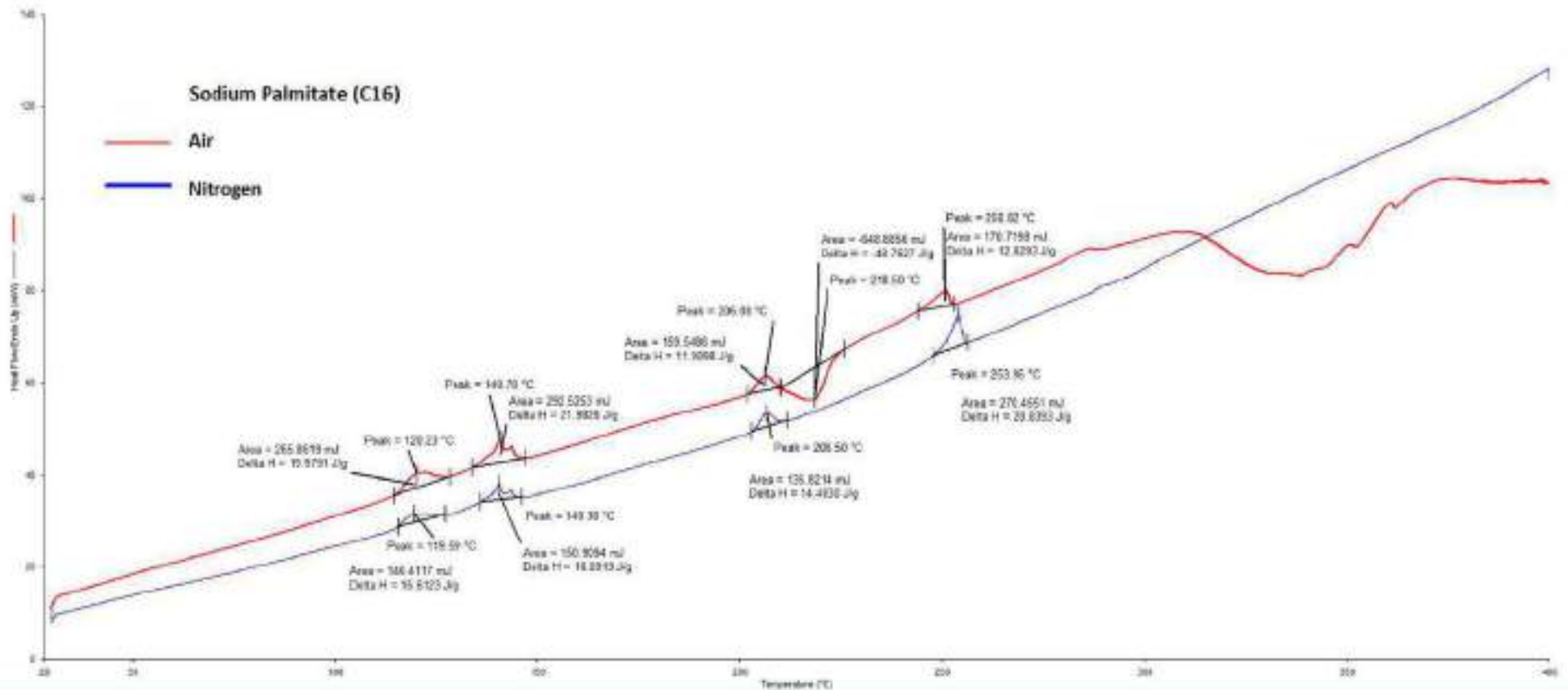
- All endothermic (requires heat)
- Each phase transition is associated to an increase of the mobility of the molecule inside the microstructure
- The first three transitions are associated to an increase of volume while in the last two the volume decreases drastically to values lower than the first one
- In AIR: oxidation at 210-225°C, exothermic. Decomposition over 300°C, exothermic

Phase change	Temperature °C
Curd fibres (omega phase) - subwaxy soap	120-126
Subwaxy soap - waxy soap	138-140
Waxy soap - subneat soap	202-206
Subneat soap - neat soap	250-259
Neat soap - isotropic liquid	283-286

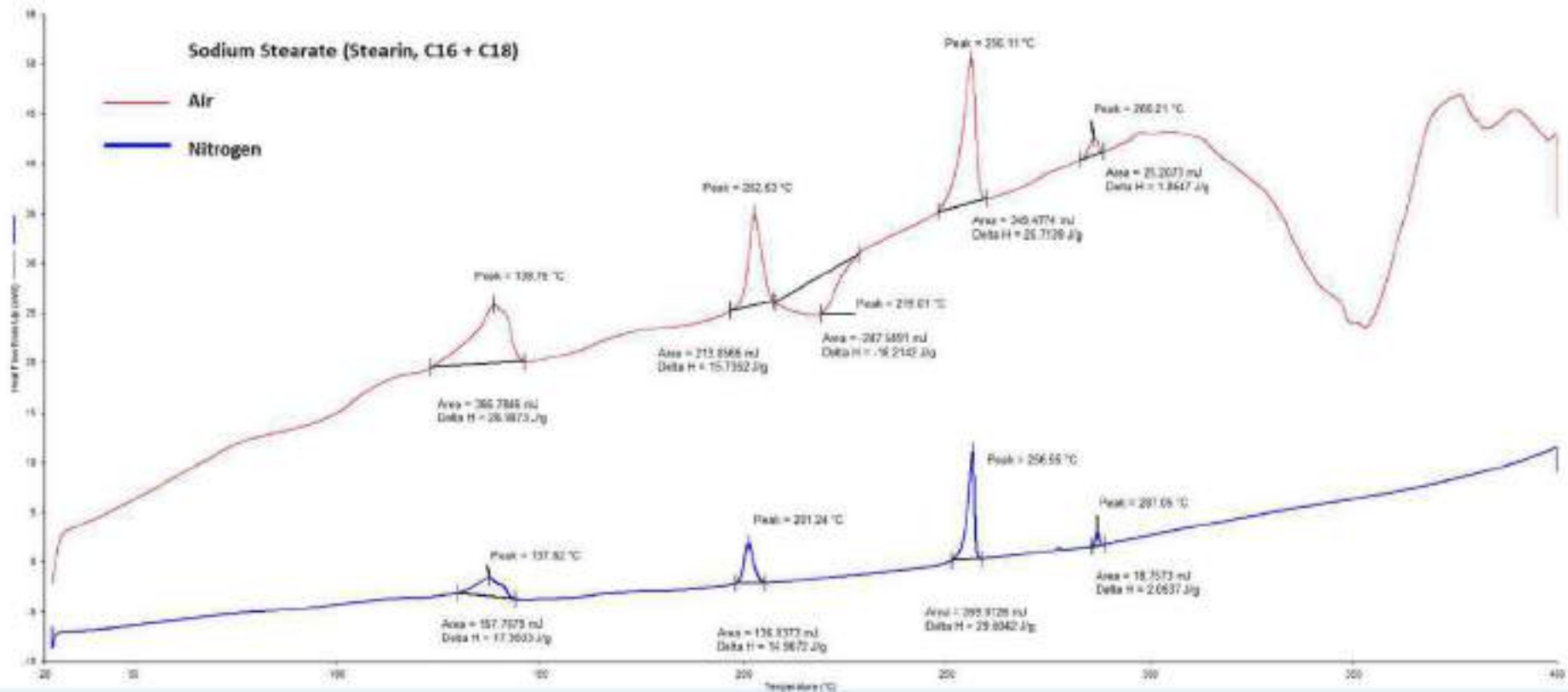
THERMAL RESISTANCE OF SODIUM SOAPS: DIFFERENTIAL SCANNING CALORIMETRY (DSC)



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THERMAL RESISTANCE OF SODIUM SOAPS: DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Sample	Peak of exothermic transition	ΔH J/g	Burning Starts
Sodium Stearate (Stearin, C16 + C18)	219	-22	320
Sodium Palmitate (C16)	218	-50	310
Sodium Stearate (Pure Stearic Acid, C18)	223	-41	350
Borax Pentahydrate	218	-35	320
Sodium Metasilicate Pentahydrate	220	-25	325
→ Melamine	//	//	315
Aluminium Hydroxide	212	-39	330
Sodium Phosphate	211	-41	300
→ Aromatic Phosphate Esther	//	//	310
Sodium Nitrate	216	-54	310
Sodium Organic Salt	239	-24	310
→ Sodium Organic Salt + Sodium Metasilicate Pentahydrate	//	//	305

THERMAL RESISTANCE OF SODIUM SOAPS: THERMOGRAVIMETRIC ANALYSIS (TGA)

- Measure variation of weight of a sample while the temperature is increased
- Thermal decomposition, oxidation, absorption, adsorption and desorption, ...

OXIDATIVE ATMOSPHERE

Atmosphere: Air

Ramp from 35°C to 700°C: 10°C/min

Pan: Alumina

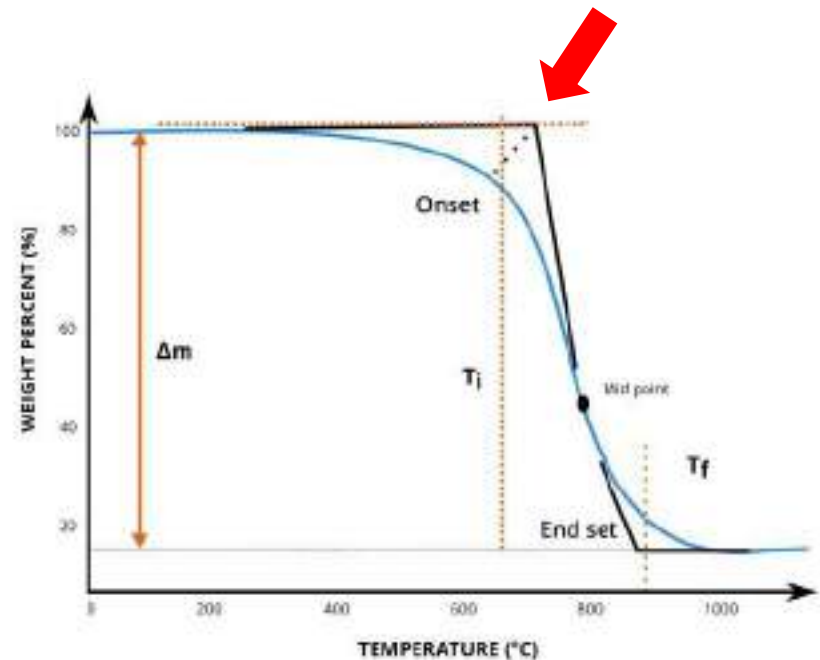
Sample weight: 15-25 mg



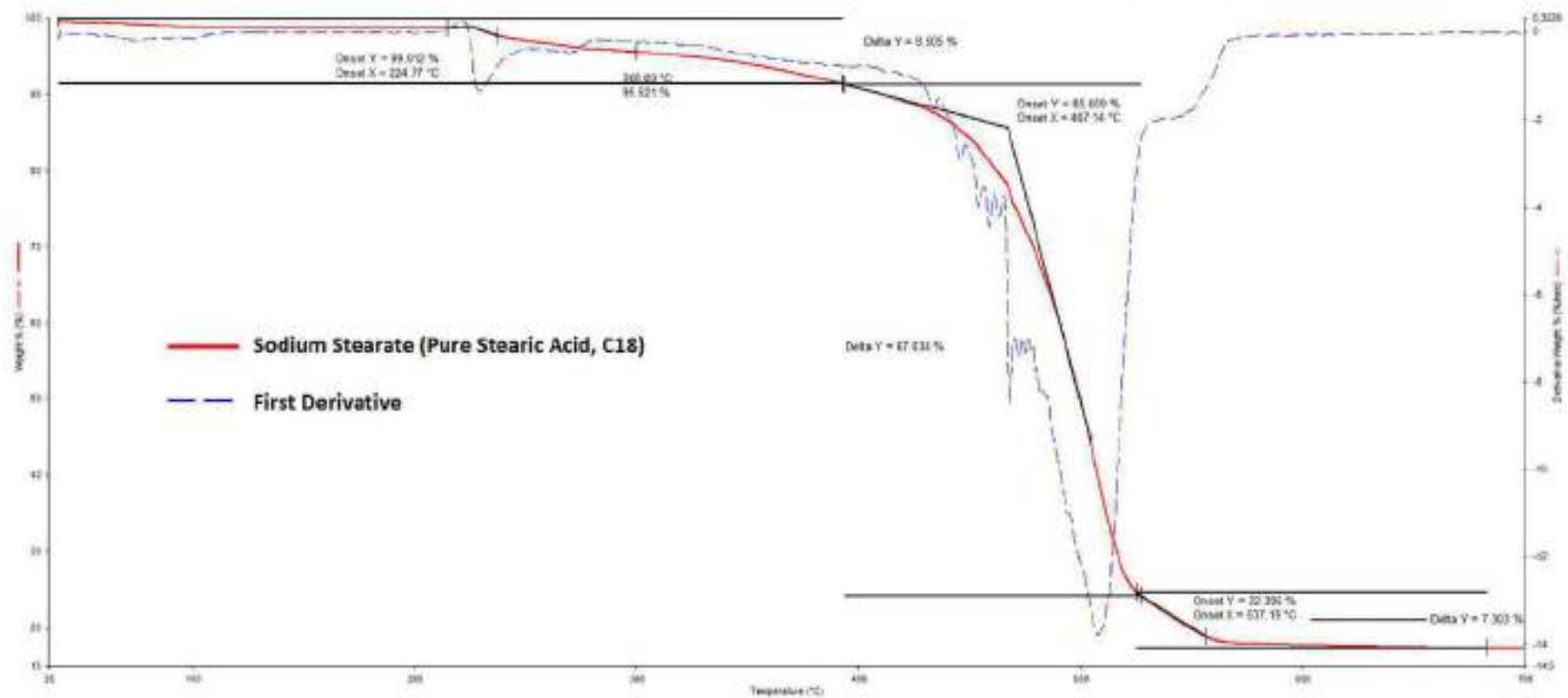
PerkinElmer TGA 4000 coupled with PolyScience chiller

THERMAL RESISTANCE OF SODIUM SOAPS: THERMOGRAVIMETRIC ANALYSIS (TGA)

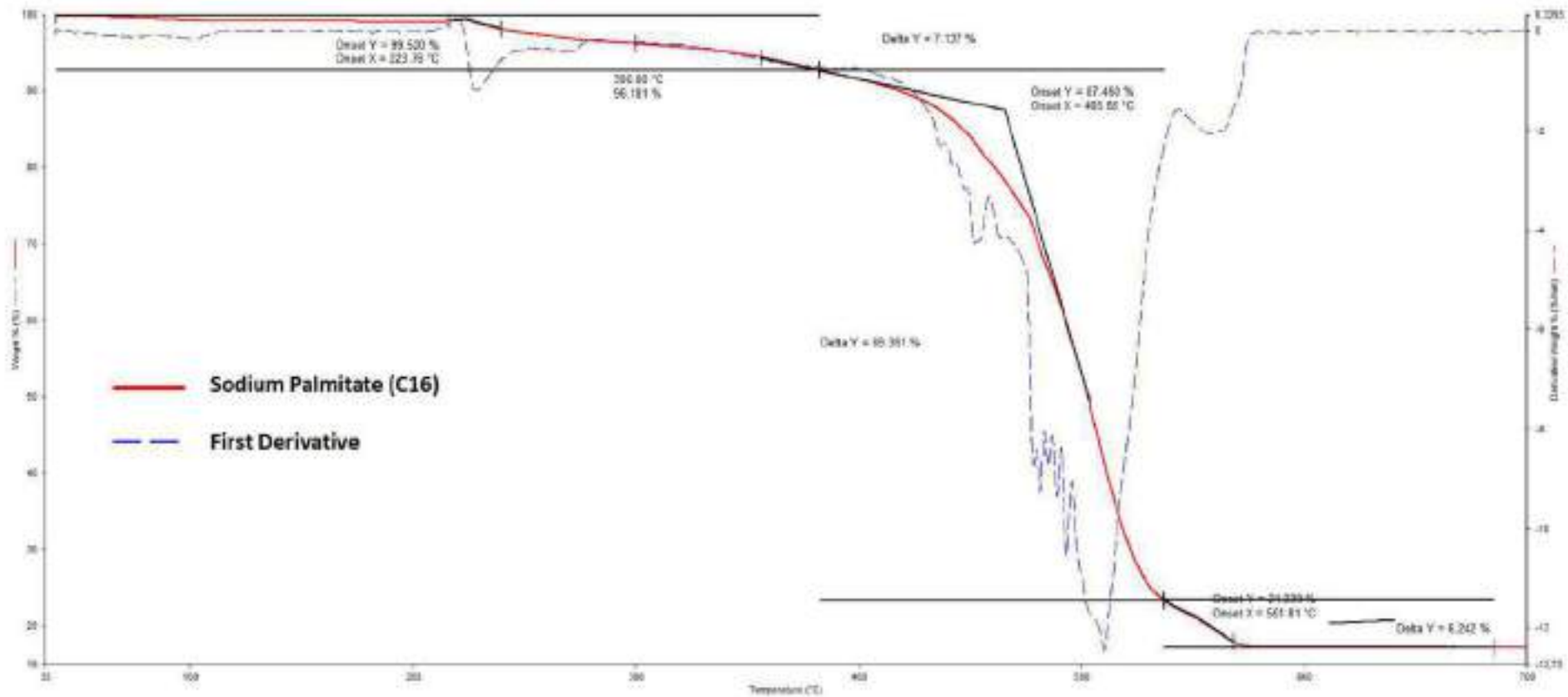
- TGA is used to evaluate the thermal stability
- The beginning of the transition is identified by the **Onset temperature** according to ASTM E2550. This is the “point in the TGA curve where a deflection is first observed from the established baseline, prior to the thermal event”
- Maximum rate of the weight loss is the inflection point of the curve and can be found through first derivative
- For soap is also useful to check the weigh loss at a fixed temperature
- Oxidative reactions produce an increase of weight



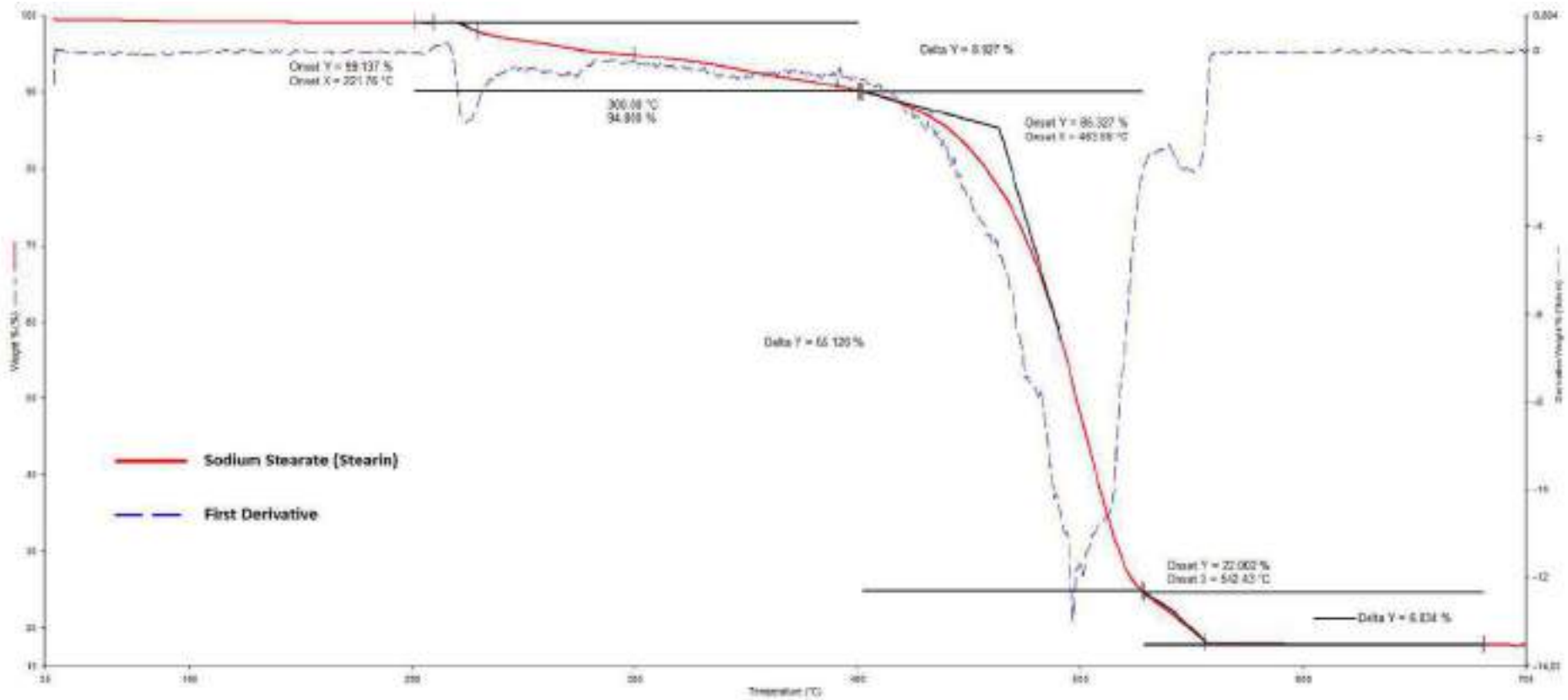
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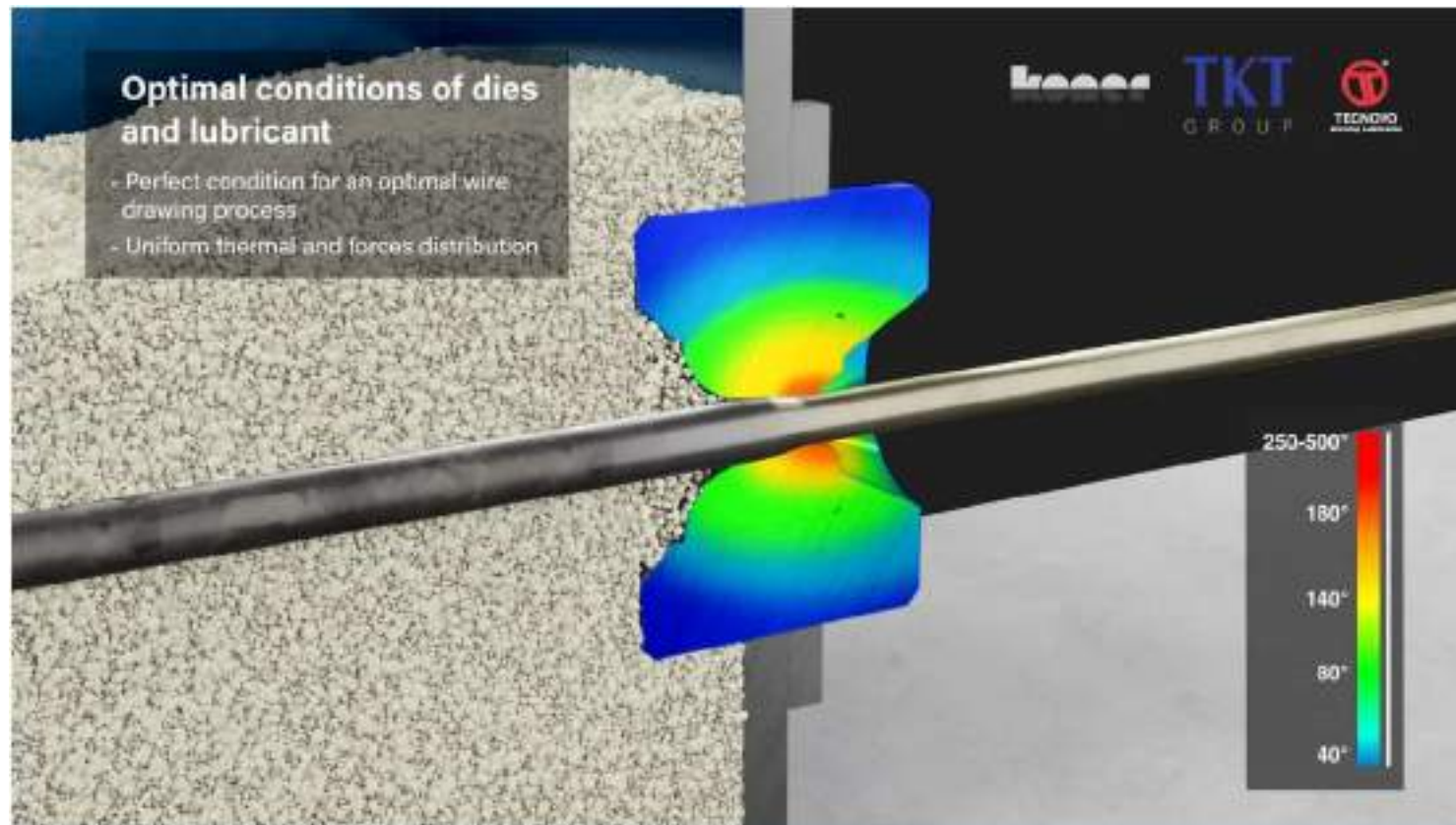


THERMAL RESISTANCE OF SODIUM SOAPS: THERMOGRAVIMETRIC ANALYSIS (TGA)

Sample	First Onset T	End	Weight loss %	Second Onset T	End	Weight loss %	Weight loss % at 300°C
Sodium Stearate (Stearin, C16 + C18)	223	425	12	425	465	83	5,5
Sodium Palmitate (C16)	221	420	12	420	463	84	3,5
Sodium Stearate (Pure Stearic Acid, C18)	225	430	11	430	467	82	4,5
Borax Pentahydrate	233	425	11,5	425	470	80	5
Sodium Metasilicate Pentahydrate	239	430	10,5	430	475	81	3,2
Melamine	224	430	15	430	473	83	4,5
Aluminium Hydroxide	222	420	13	420	460	81	6
Sodium Phosphate	230	410	9	410	450	81	3
Aromatic Phosphate Esther	245	430	14	430	461	82	4,7
Sodium Nitrate	235	380	9	425	462	81	1,8
Sodium Organic Salt	250	450	13	450	480	80	1,8
Sodium Organic Salt + Sodium Metasilicate Pentahydrate	270	435	8	435	472	79,5	1,5

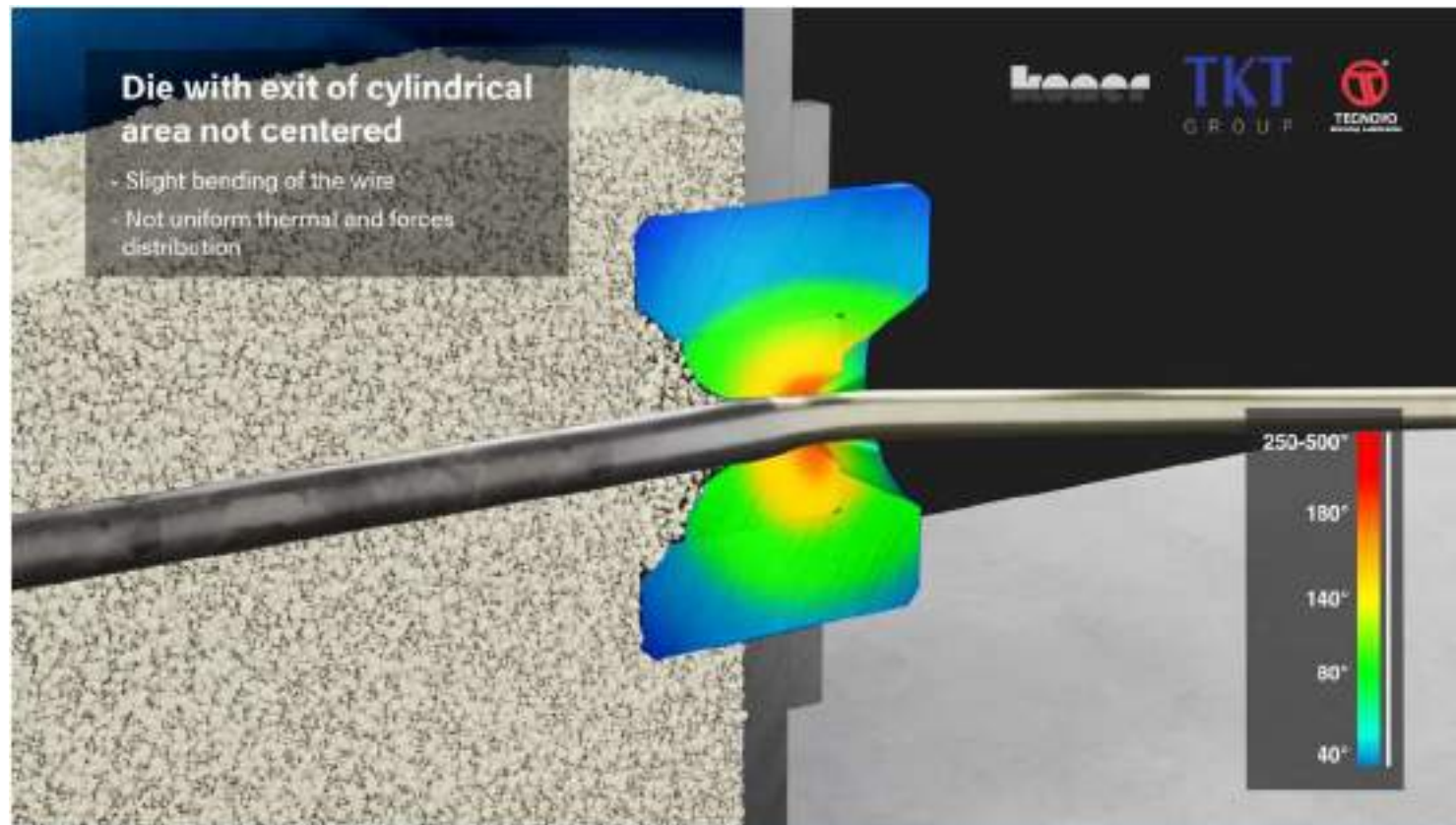
HOW LUBRICANTS AND DIES INFLUENCES THE DRAWING PROCESS

Optimal conditions of die and lubricant



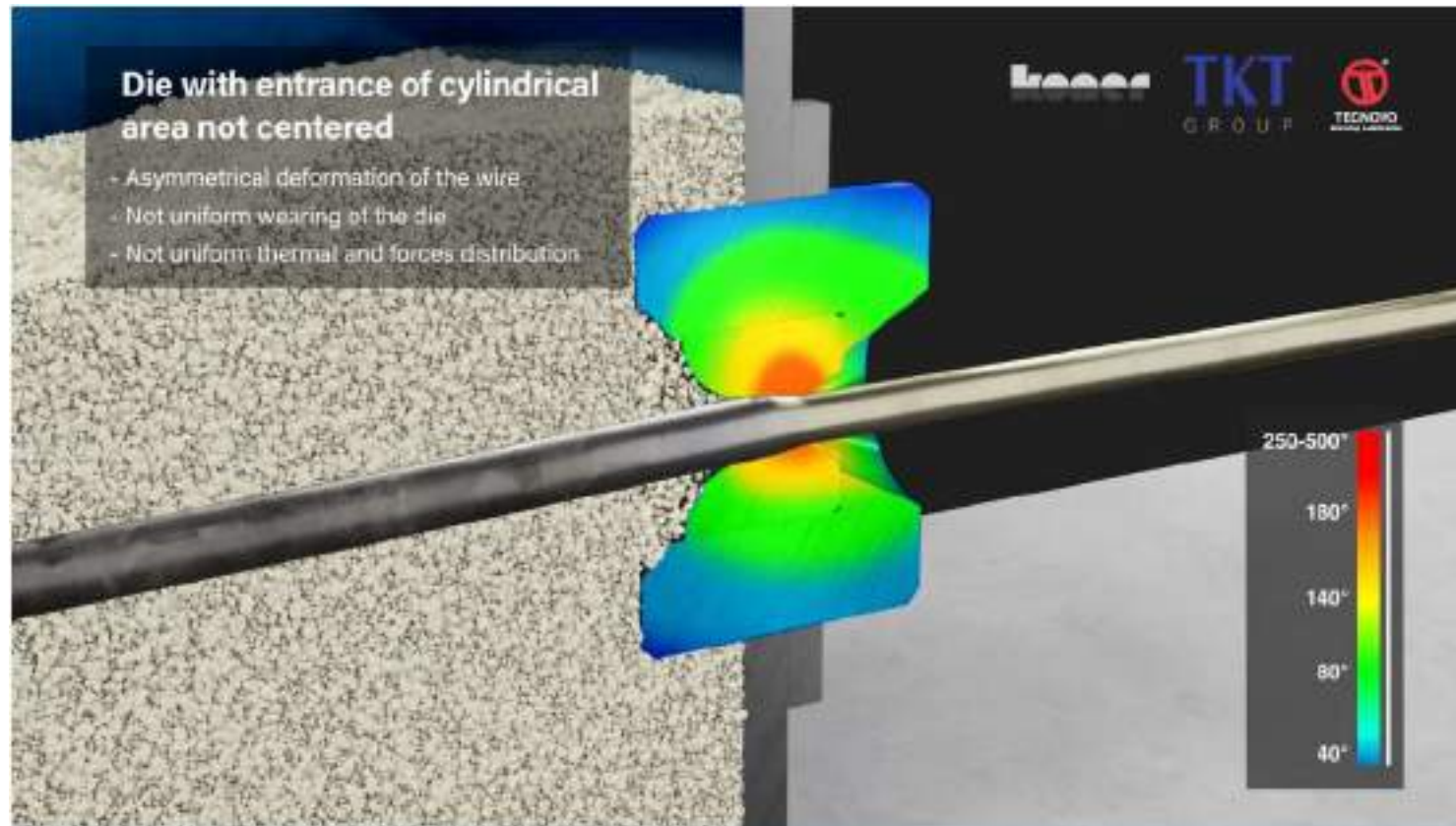
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Die with exit of cylindrical area not centered



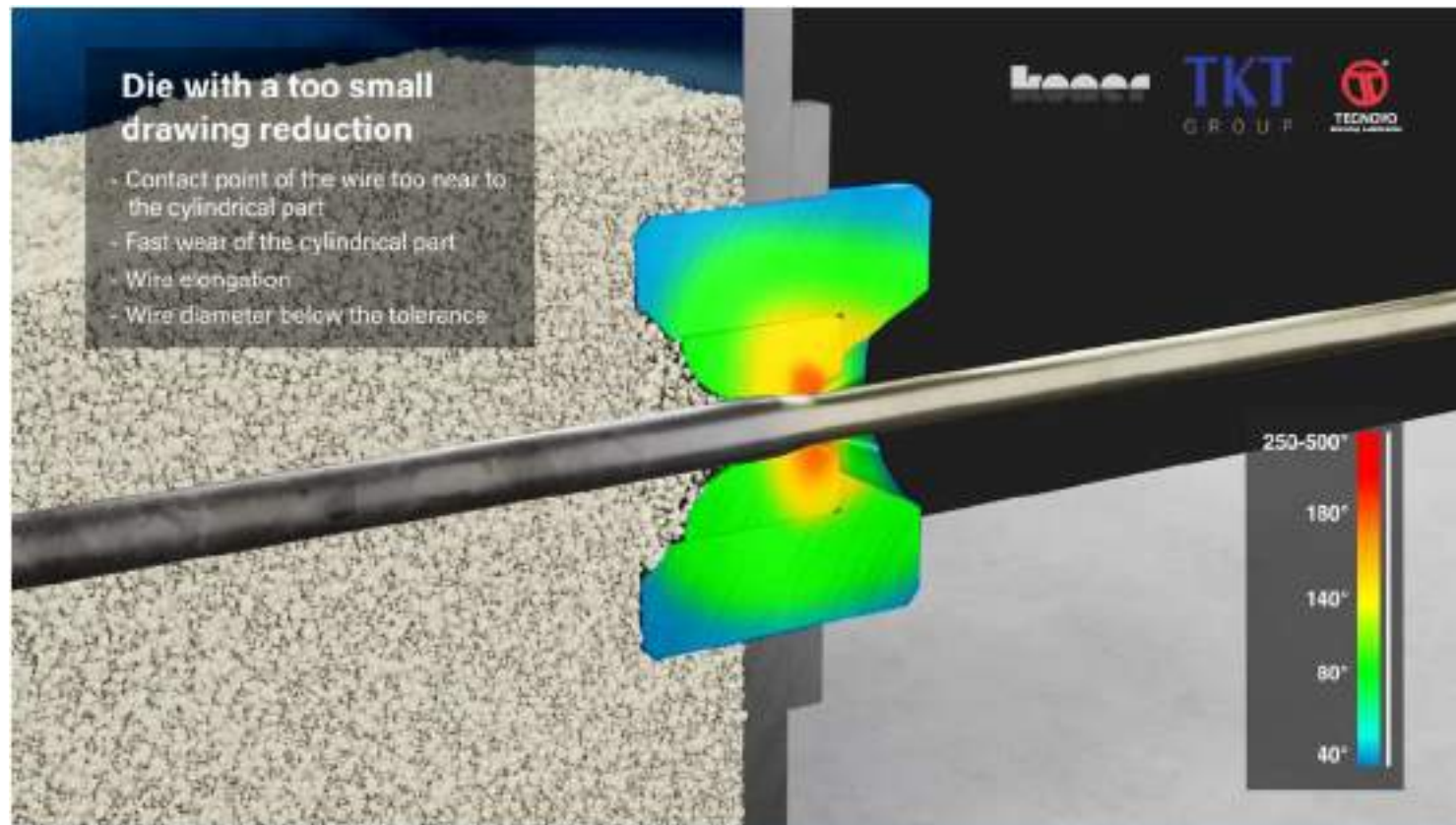
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Die with entrance of cylindrical area not centered



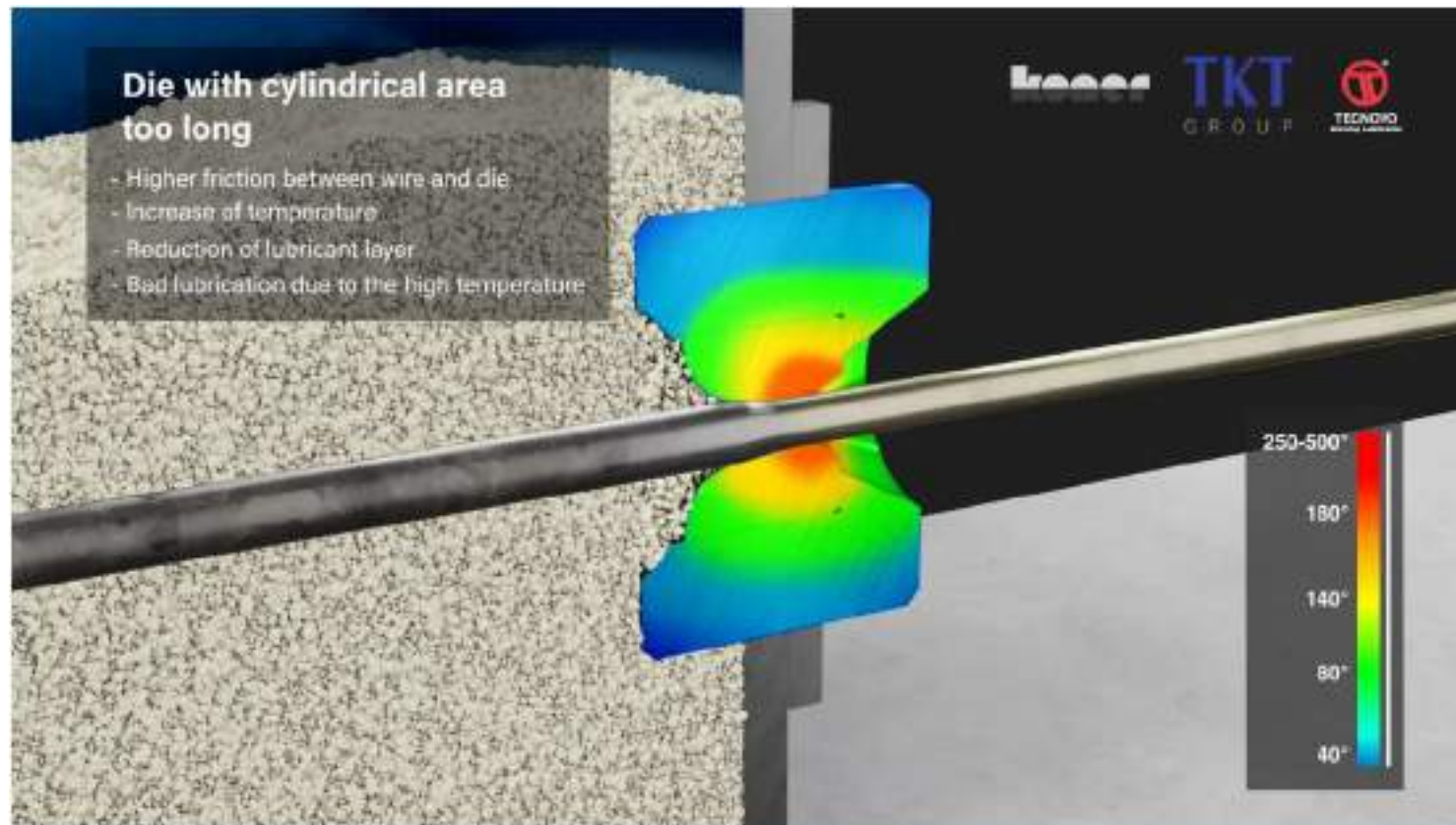
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Die with a too small drawing reduction



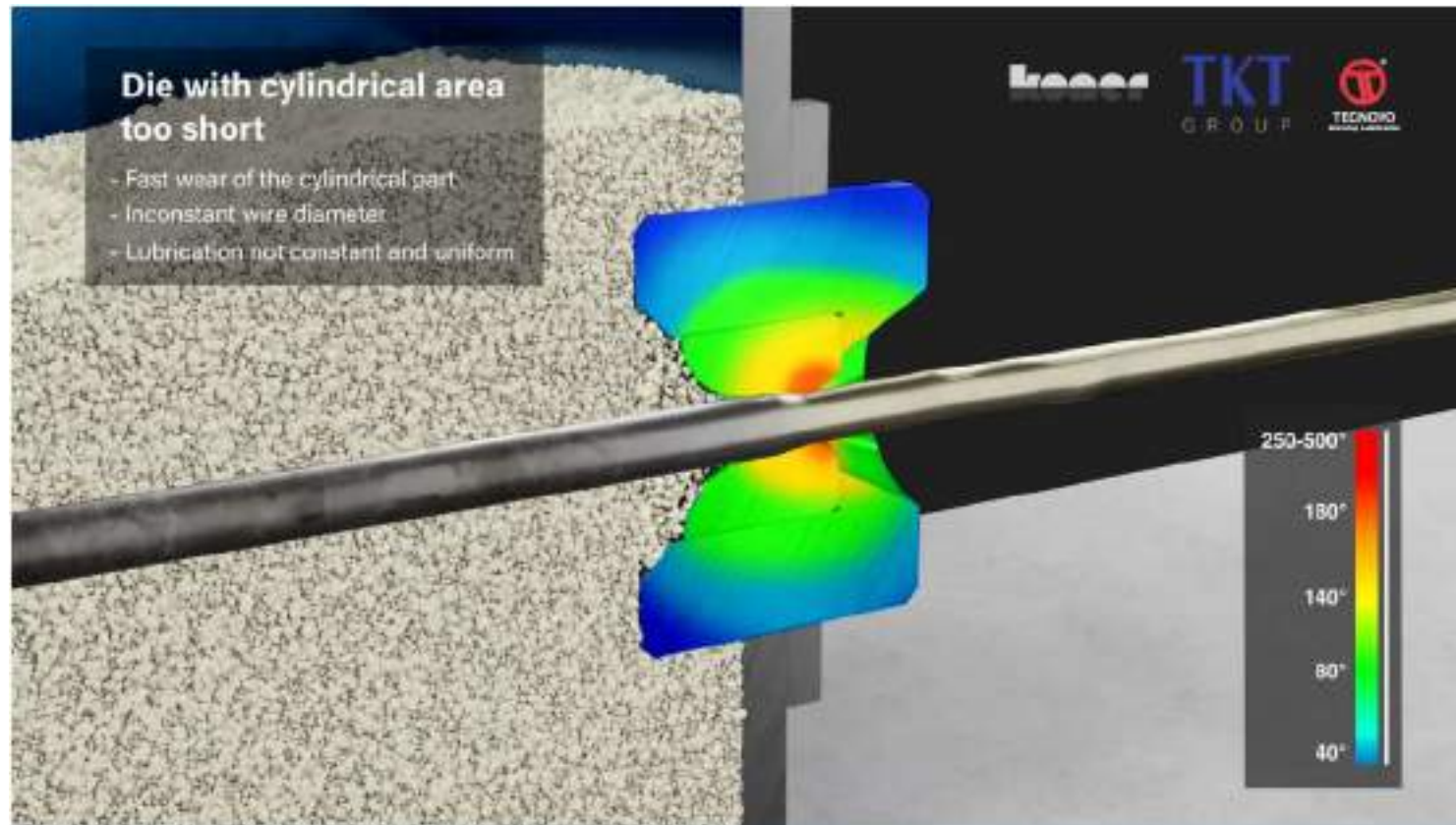
HOW LUBRICANTS AND DIES INFLUENCES THE DRAWING PROCESS

Die with cylindrical area too long



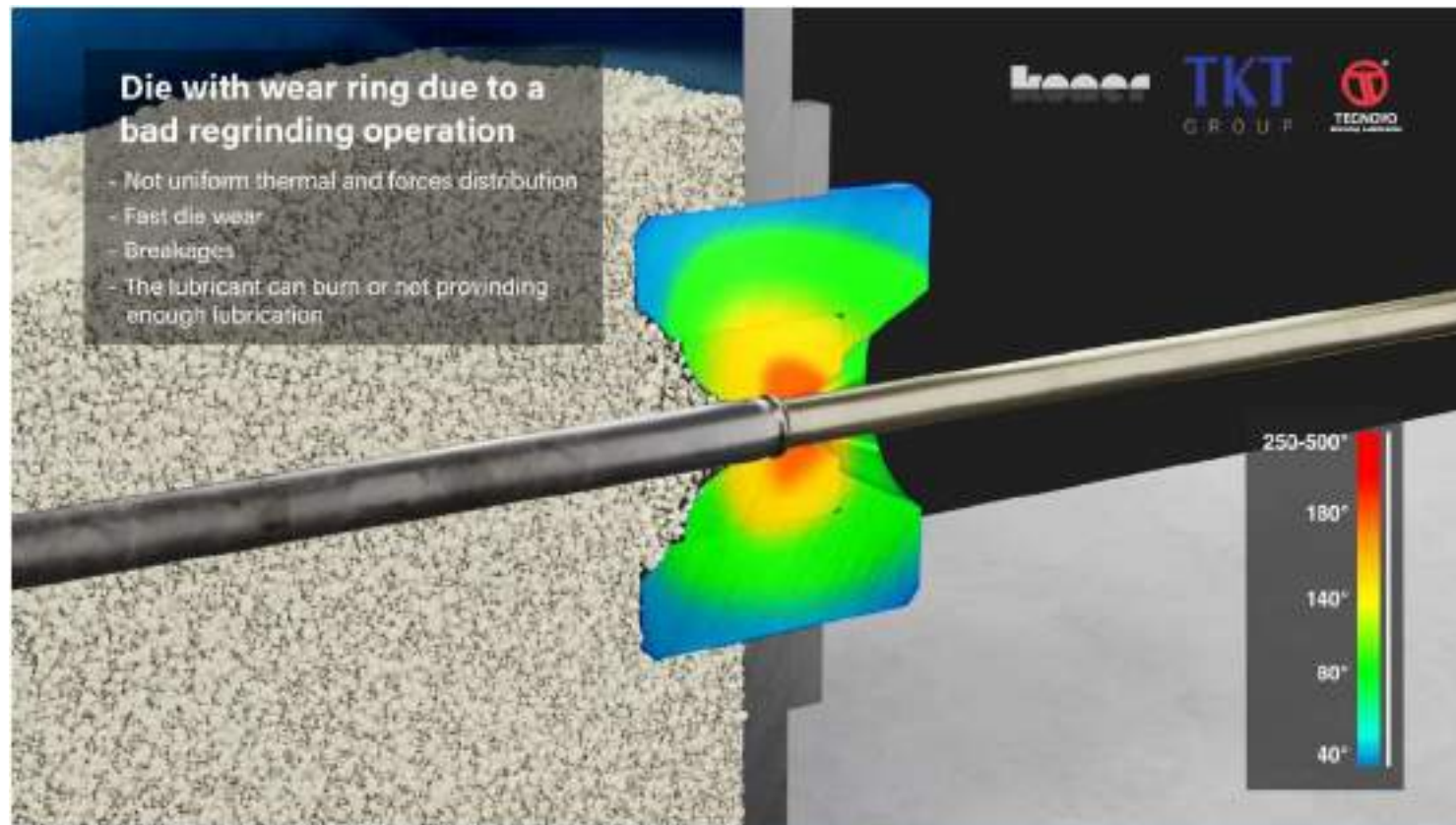
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Die with cylindrical area too short



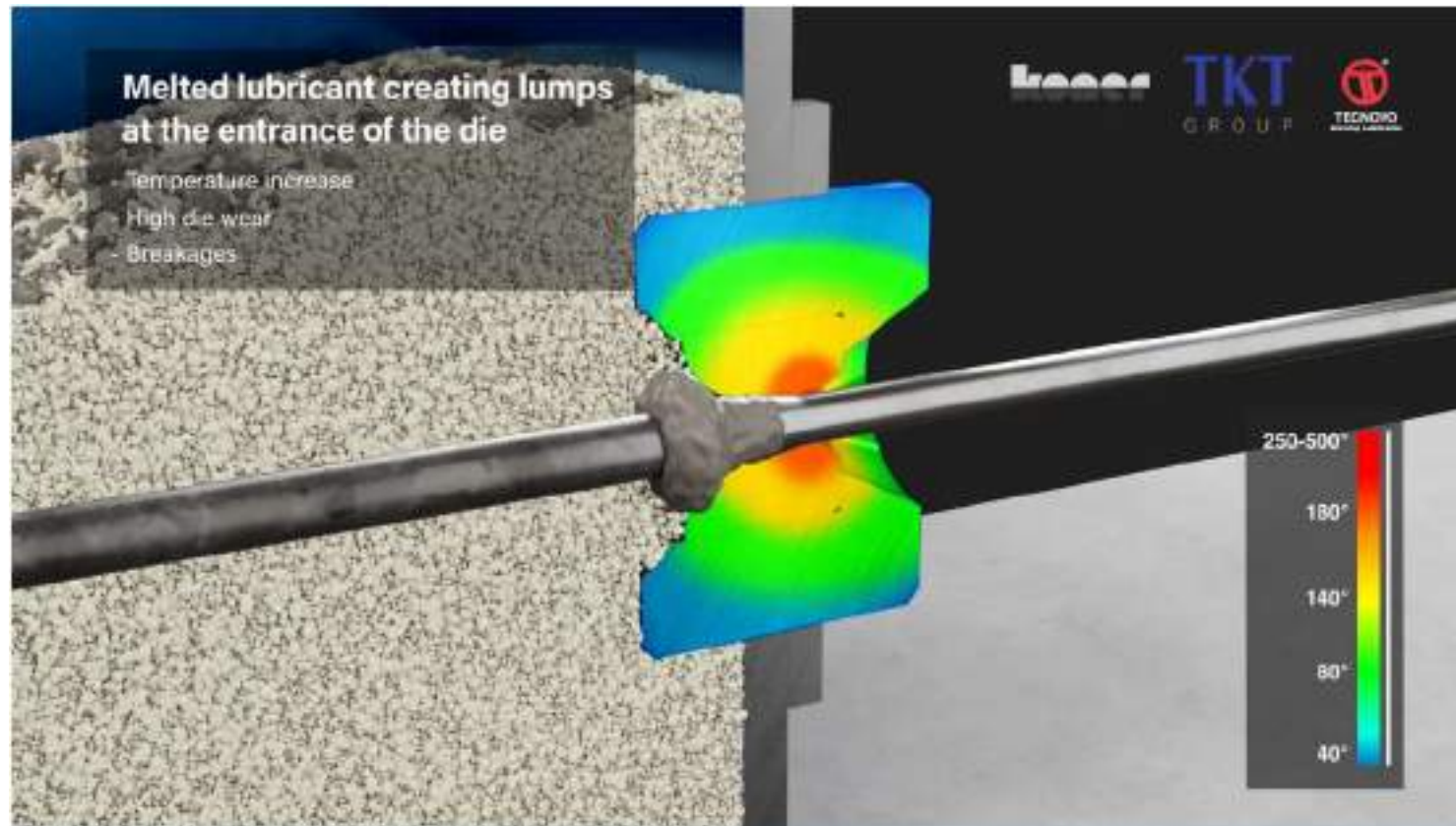
HOW LUBRICANTS AND DIES INFLUENCES THE DRAWING PROCESS

Die with wear ring due to a bad regrinding operation



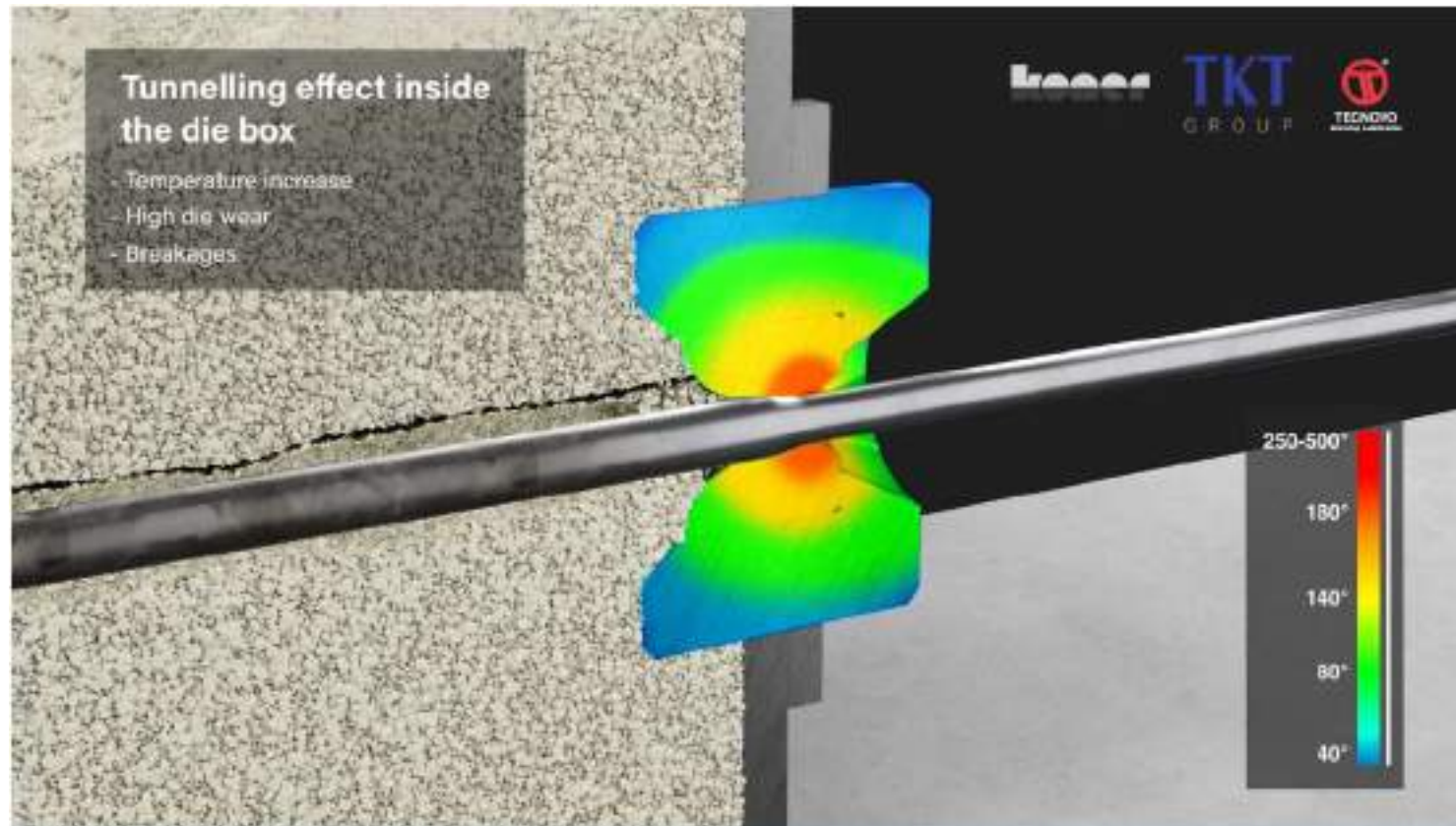
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Melted lubricant creating lumps at the entrance of the die



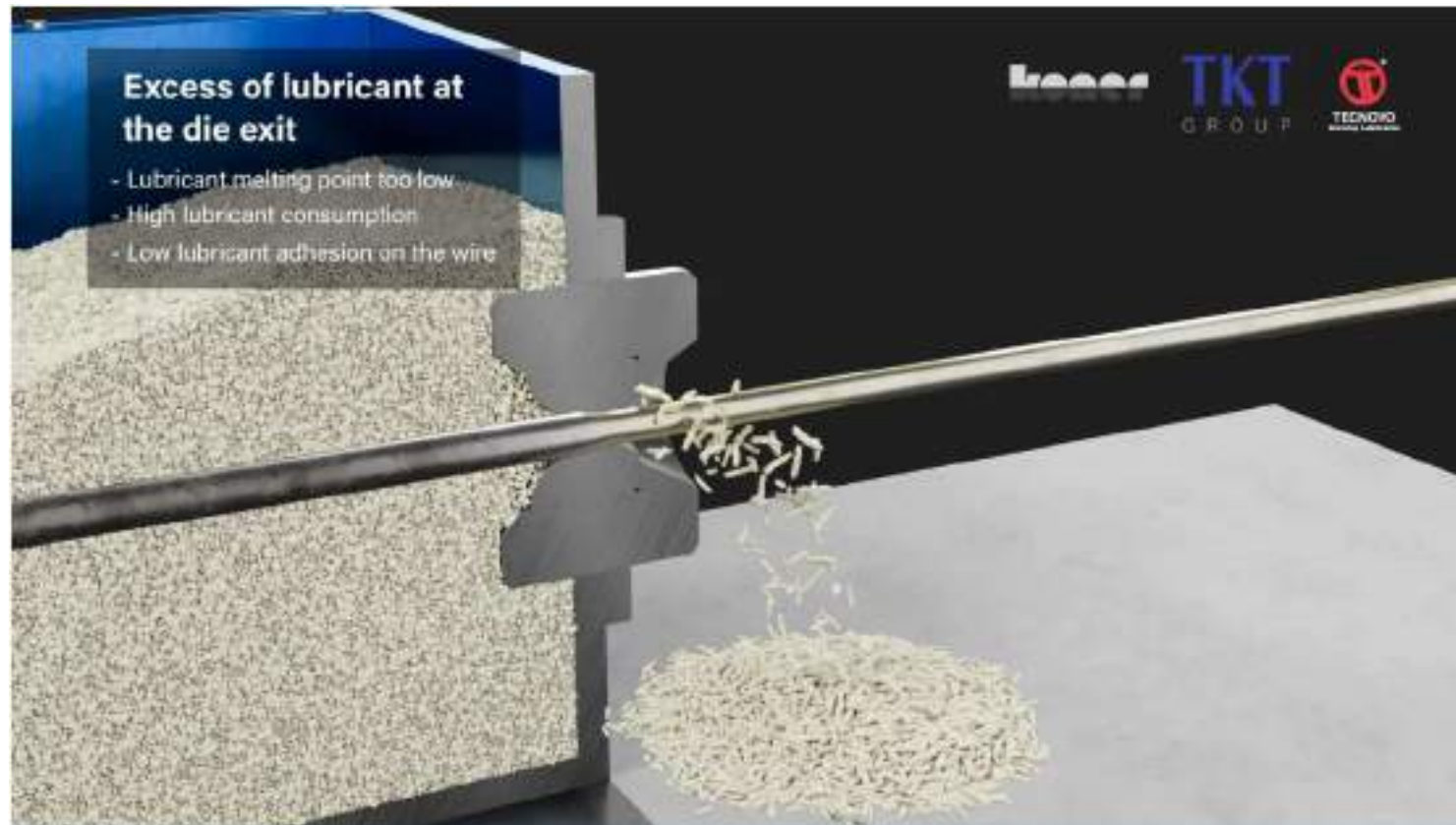
HOW LUBRICANTS AND DIES INFLUENCES THE DRAWING PROCESS

Tunnelling effect inside the die box



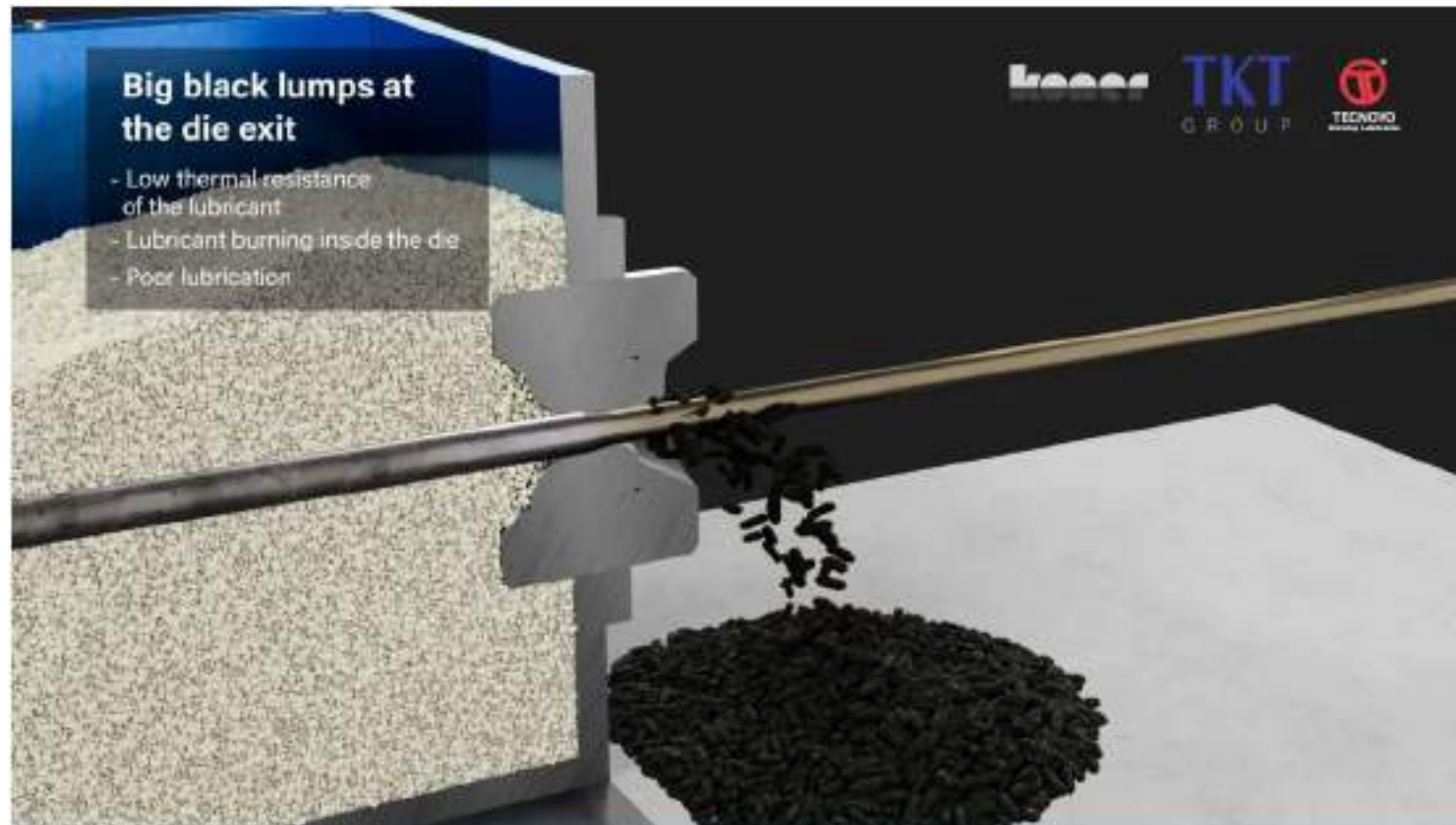
HOW LUBRICANTS AND DIES INFLUENCES THE DRAWING PROCESS

Excess of lubricant at the die exit



HOW LUBRICANTS AND DIES INFLUENCES THE DRAWING PROCESS

Big black lumps at the die exit





CASE HISTORY: PRODUCTION OF VERY BRIGHT WIRE





THANK YOU FOR YOUR KIND ATTENTION



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