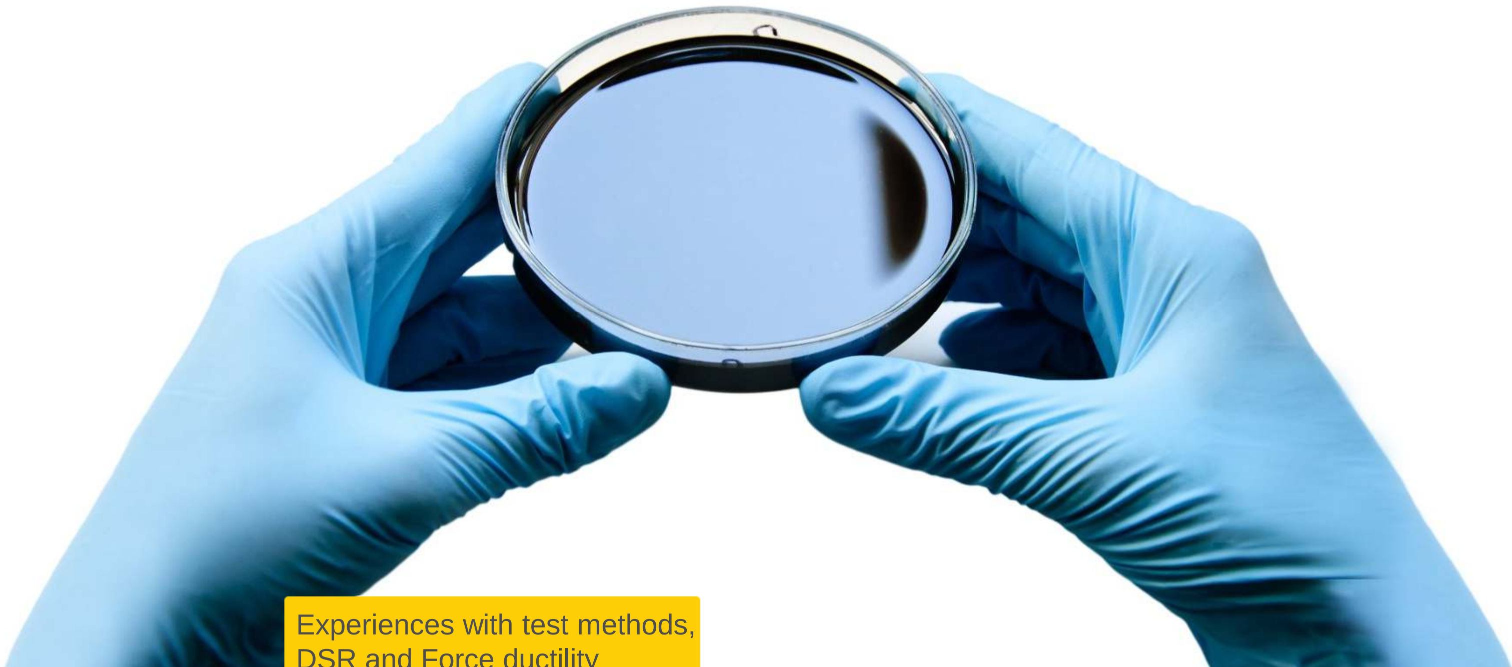


Experiences with test methods,
DSR and Force ductility

Carl Hultin

Nynas experience with DSR and Force ductility

- Long experience with this testing
- DSR
  - The impact of sample preparation on the rheology of PmBs
    - Sample preparation methods
    - Influence on PmB type
  - The EN-standard
  - N200 specification
- Force Ductility
  - Binder cohesion and polymer impact on cohesion
  - Requirement for PMB in new N200 specification

Handbook N200 Vegbygging

(Handbook N200 Road Construction)

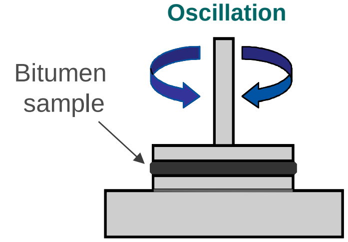
- New requirements for PmBs
- Force ductility at 10°C
  - Only TBR in previous version
- Additional requirements for DSR
  - Does not apply generally, only where this is specified in the contract
  - Can be used if a contractor want a more specified PmB
    - Temperature when G*=15kPa and 5 000 kPa
    - MSCR at 60°C
- Norway is at the forefront

Tabell 651.3 Krav til polymermodifisert bitumen

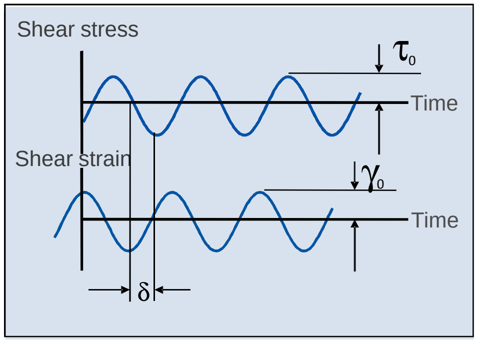
	Enhet	Provningsmetod NS-EN	Gradering				
			65/105-60	40/100-75	90/150-60	75/130-80	25/55-75
Penetrasjon ved 25°C	0,1 mm	1426	65-105	40-100	90-150	75-130	25-55
Kohesjon målt med kraftduktilitet	**J/cm^2**	**13589**	**≥ 1 ved 10°C**	**≥ 2 ved 10°C**	**≥ 0,5 ved 10°C**	**≥ 2 ved 10°C**	**≥ 3 ved 10°C**
Frass bruddpunk	°C	12593	≤ -12	≤ -12	≤ -18	≤ -20	≤ -10
Elasisk tilbakegang ved 10°C	%	13398	≥50	≥75	≥75	≥75	≥50
Flammepunk	°C	ISO 2592	≥220	≥220	≥220	≥220	≥220
Lagringsstabilitet 72 timer ved 180°C		13399	Krav til lagringsstabilitet				
Forsell i mykningspunkt	°C	1427	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
Forsell i penetrasjon	0,1 mm	1426	≤ 9	≤ 9	≤ 9	≤ 9	≤ 9
			Krav til gjenværende egenskaper etter korttidsaldring				
Motstand mot opphërdning, RTFOT ved 163°C		12607-1					
Masseendring	%	12607-1	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5
Gjenværende penetrasjon	%	1426	≥60	≥60	≥60	≥60	≥60
Økning i mykningspunkt	°C	1427	≤ 10	≤ 10	≤ 10	≤ 10	≤ 10
Fall i mykningspunkt	°C	1427	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
Ytelserelaterte tilleggskrav Gjelder ikke generelt, kun der dette er spesifisert i kontrakten.							
Motstand mot opphërdning, RTFOT ved 163°C		12607-1	Krav til gjenværende egenskaper etter korttidsaldring				
MSCRT Jnr3,2 kPa ved 60°C	**kPa^{-1}**	**16659**	**≤ 0,5**	**≤ 0,2**	**≤ 1,0**	**≤ 0,2**	**≤ 0,1**
Temperatursensitivitet T for G*=15 kPa @ 1,59 Hz	**°C**	**14770 med 25 mm plate**	**≥50**	**≥55**	**≥40**	**≥55**	**≥60**
Temperatursensitivitet T for G*=5000kPa @ 1,59 Hz	**°C**	**14770 med 8 mm plate**	**≤ 20**	**≤ 25**	**≤ 15**	**≤ 25**	**≤ 30**
timer							
BBR etter langtidsaldring T (S=300 Mpa)	°C	14771	≤ -15	≤ -15	≤ -21	≤ -24	≤ -12

Dynamic Shear Rheometer (DSR)

- Analyse visco-elastic behaviour
  - Stiffness, viscosity, elasticity as a function of loading time and temperature
  - Sample amounts are small (~0,5g)
- Experience in US for almost 30 year
  - PG-classification (e.g PG 64-22)
- On it’s way into the PMB specification EN-14023
  - Temperature at a specific G*
  - MSCR



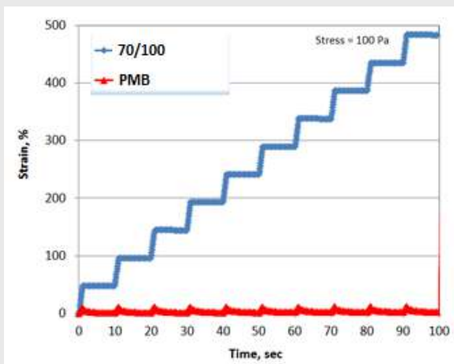
$$G^* = \frac{\tau_A}{\gamma_A}$$



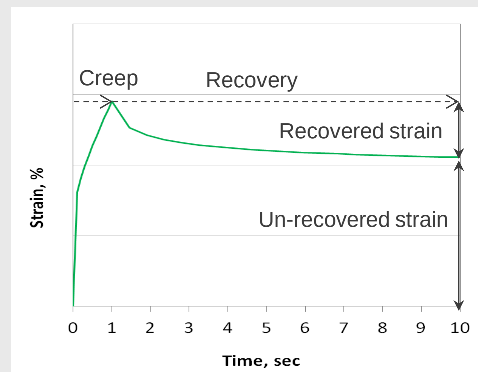
Anton Paar, DSR

Multiple Stress Creep and Recovery Test (MSCR)

- Resistance to permanent deformation - Correlation with wheel tracking



- 10 cycles of creep & recovery
- Two stress levels; 100Pa and 3200Pa
- Average strain recovery
- Average Jnr



- 1 cycle
- $\text{Strain recovery \%} = \dfrac{\text{Recovered strain}}{\text{Peak strain}} \times 100$
- $\text{Non-recoverable creep compliance } (J_{nr}) = \dfrac{\text{Unrecovered strain}}{\text{Applied stress}}$
- Jnr is expressed in [1/kPa]

Sample preparation DSR

The EN-standard allows a wide interpretation for sample preparation

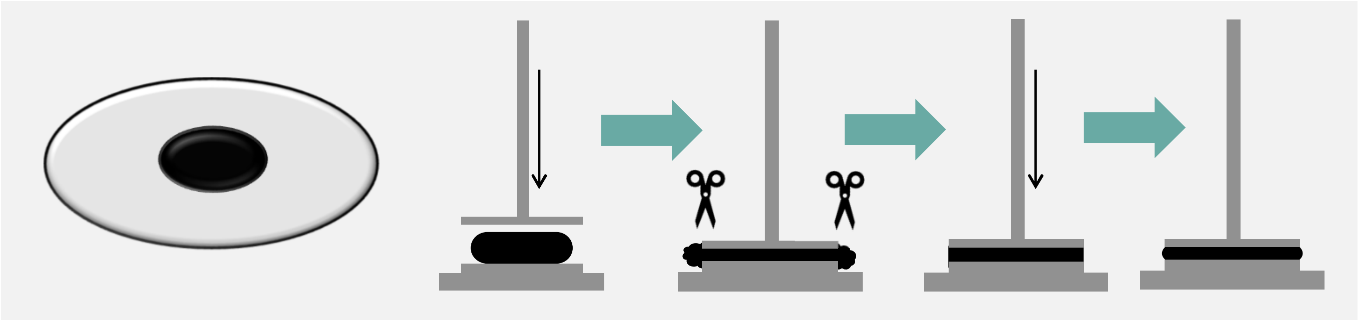
- Examples
  - Directly pour the binder on the plates
  - Pour the binder in a mould, then place the sample on the plate
  - Use vials
- Samples can be trimmed, but not obligatory
- Many laboratories have developed their own way of preparing samples

Two preparation method will be compared:

- The trimming method
  - Excess binder is squeezed between plates, excess binder is trimmed
  - This procedure might change the polymer network and morphology
- Thin mould method
  - Required amount of binder is poured in a thin adapted mould
  - Trimming is not needed
  - The polymer network doesn't change much when mounted between plates

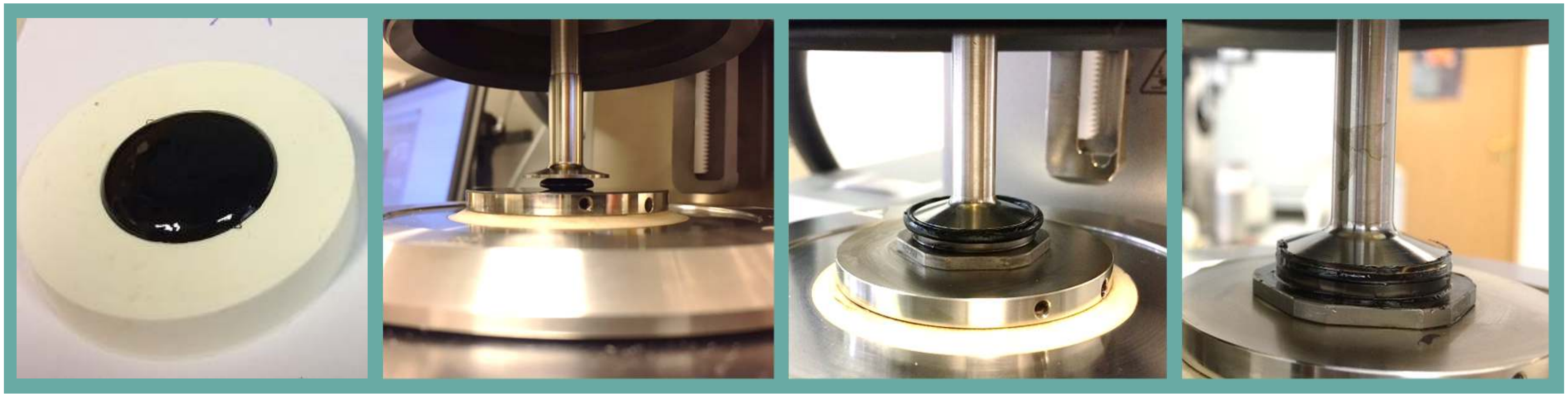
Sample preparation DSR

Trimming

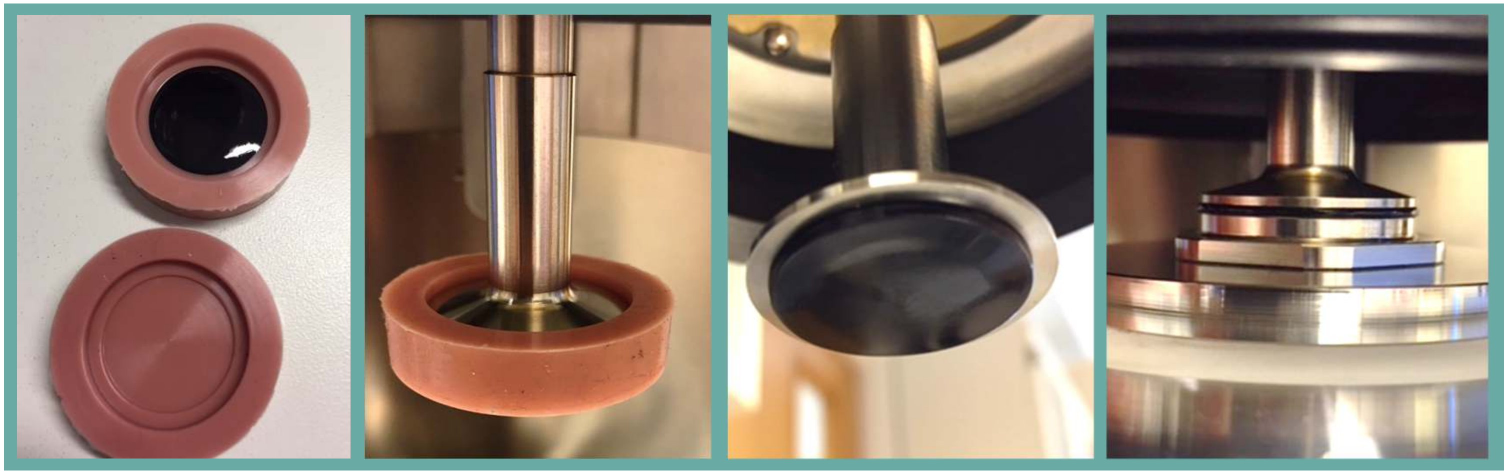


Thin mould

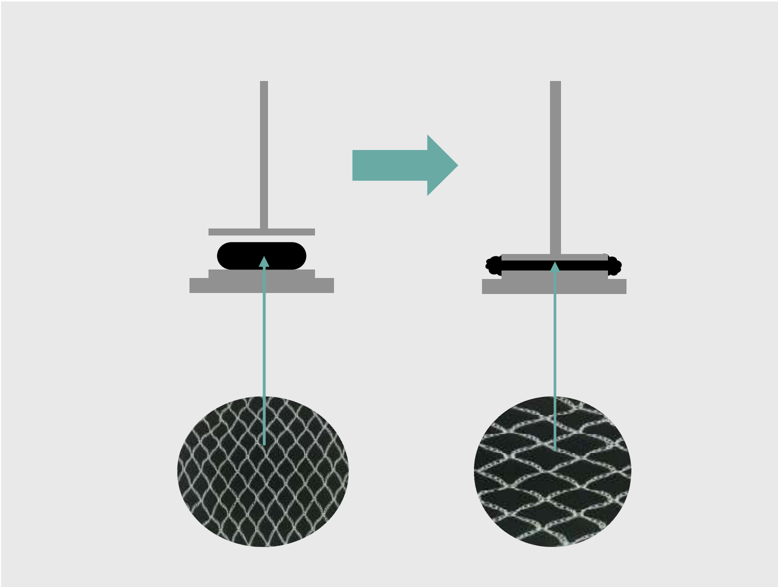
Trimming



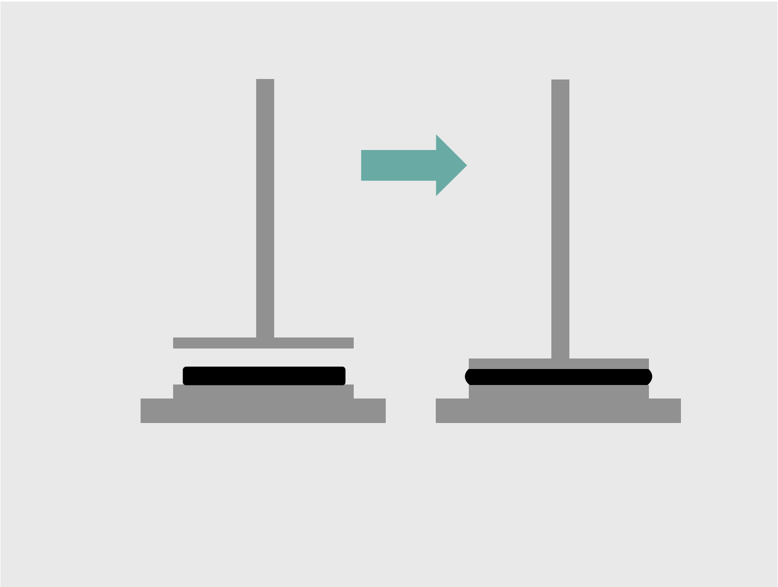
Thin mould



- The squeezing can change the morphology
  - It can be compared to a net that is stretch out



- The thin mould effects the morphology as little as possible



Experimental

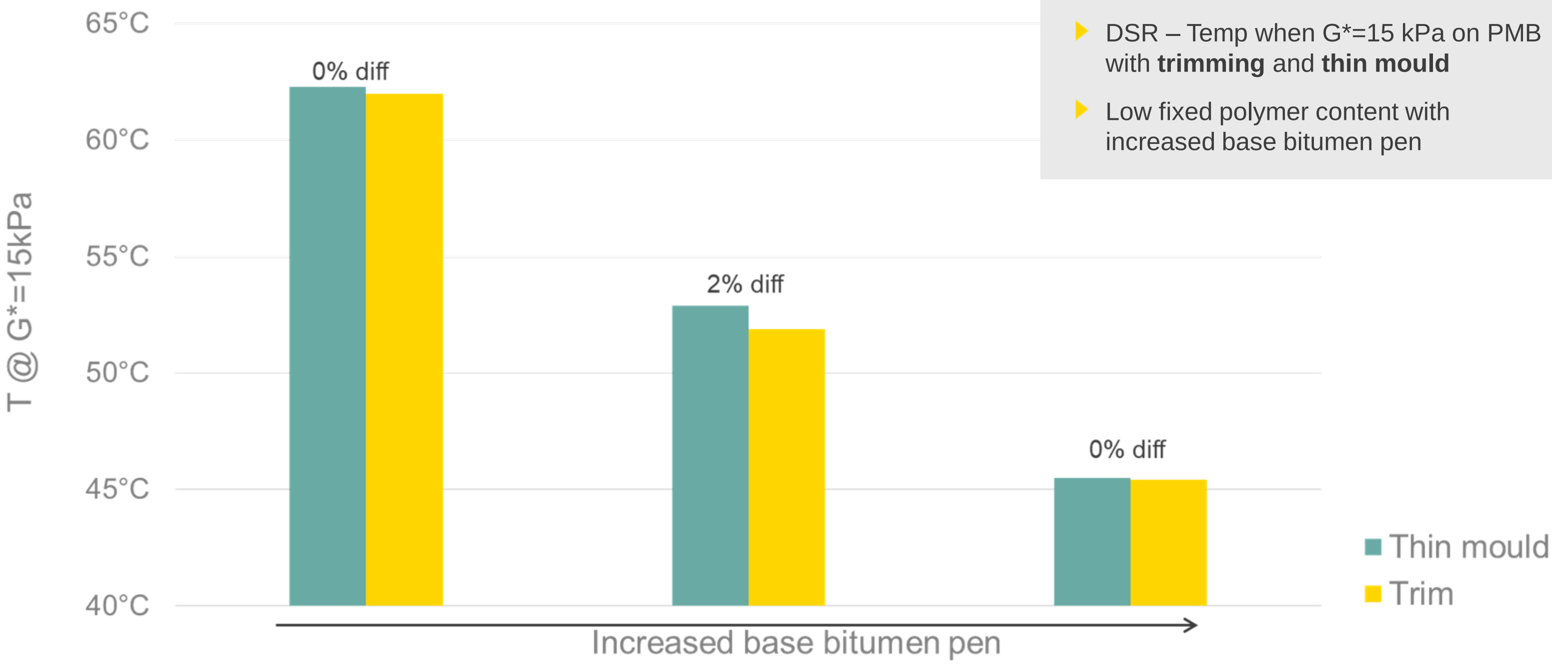
- PMBs with different polymer content and different base binder hardness

- DSR with the two preparations method (thin mould and trimming)
  - Temp when G*=15 kPa
  - MSCR at 60°C
  - DSR & MSCR tested after RTFOT

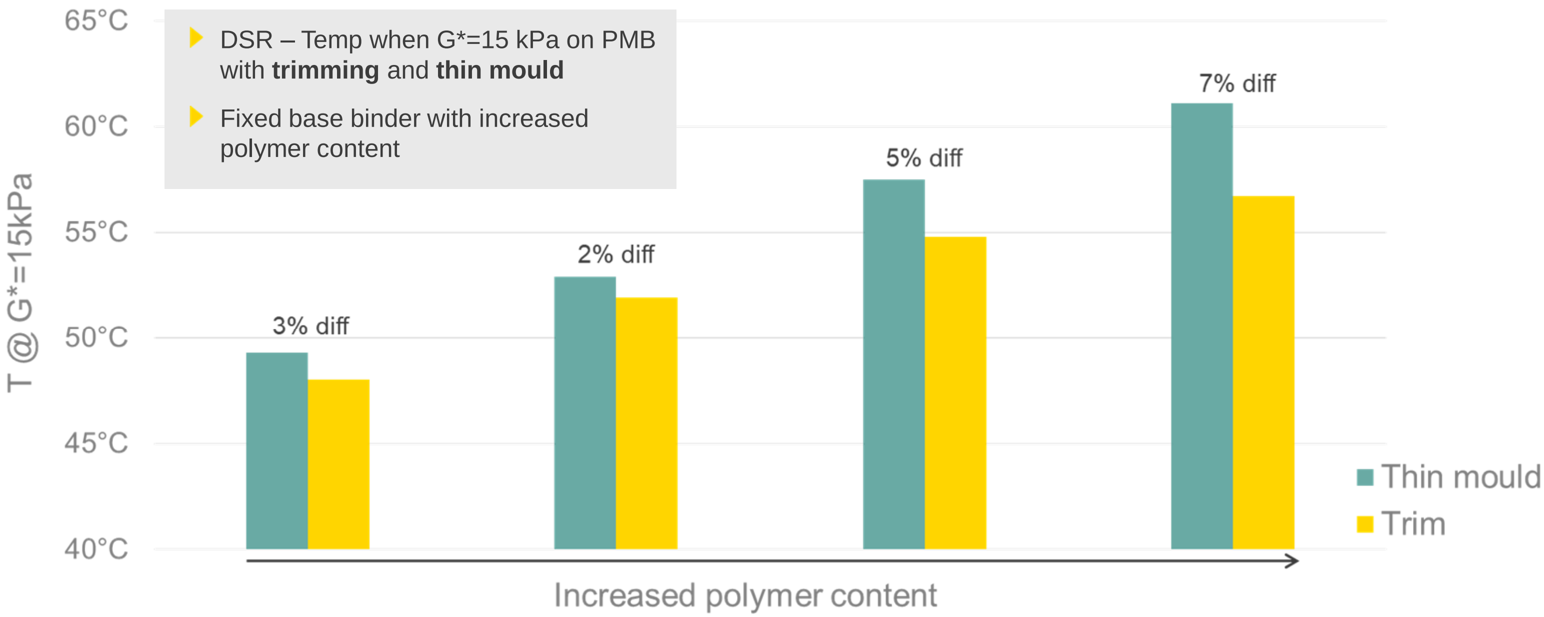
- Softening point

Thin mould vs trimming

- DSR – Temp when G*=15 kPa on PMB with **trimming** and **thin mould**
- Low fixed polymer content with increased base bitumen pen

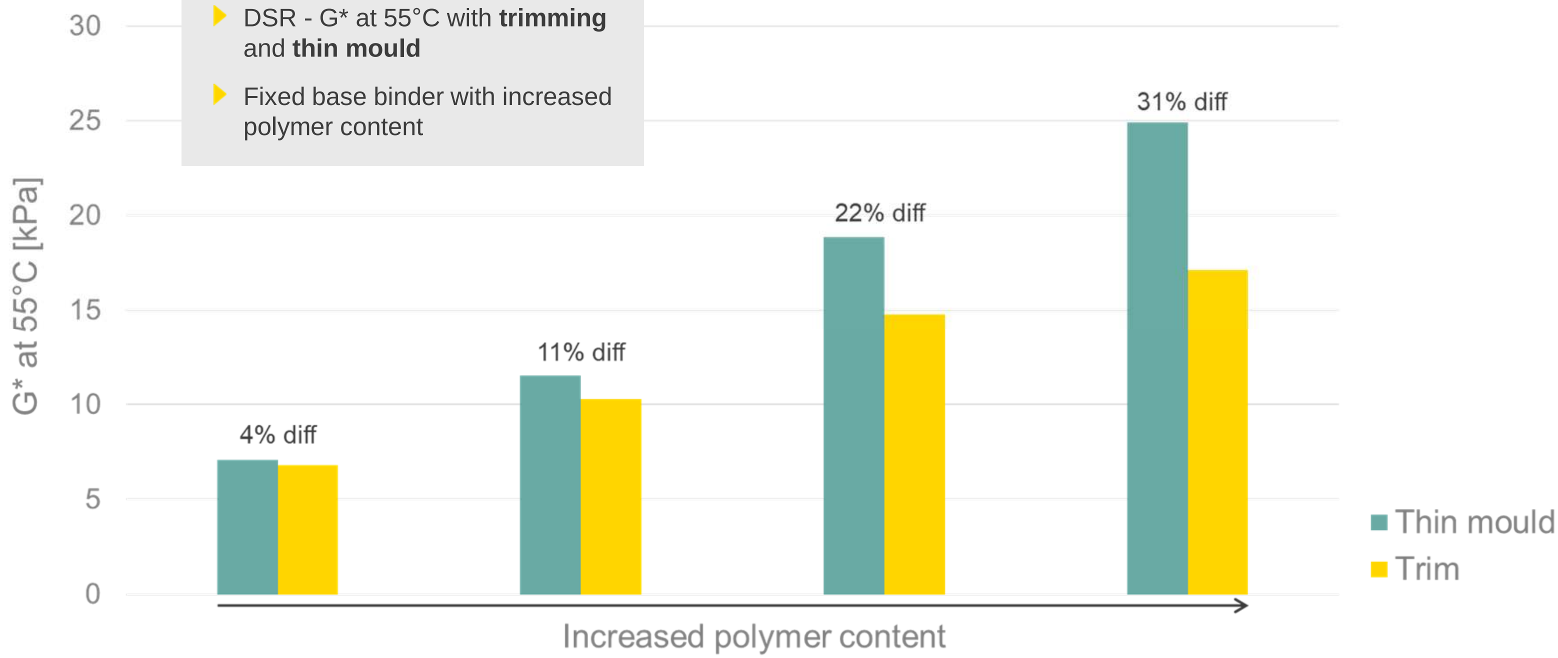


Thin mould vs trimming



Thin mould vs trimming

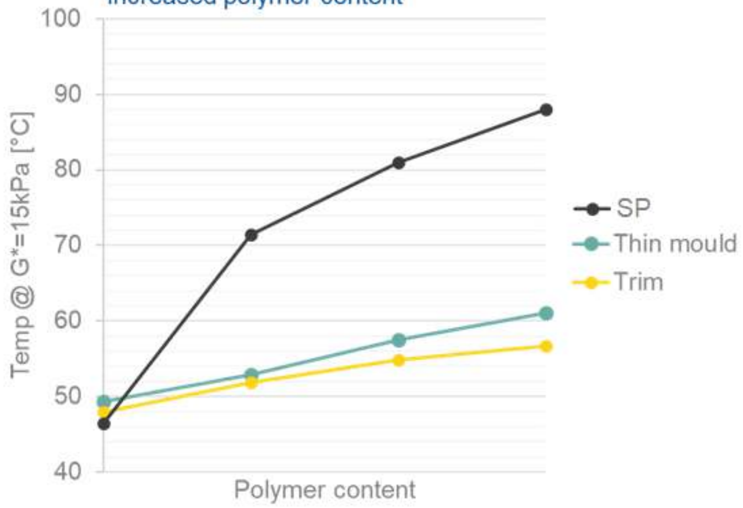
- DSR - G* at 55°C with **trimming** and **thin mould**
- Fixed base binder with increased polymer content



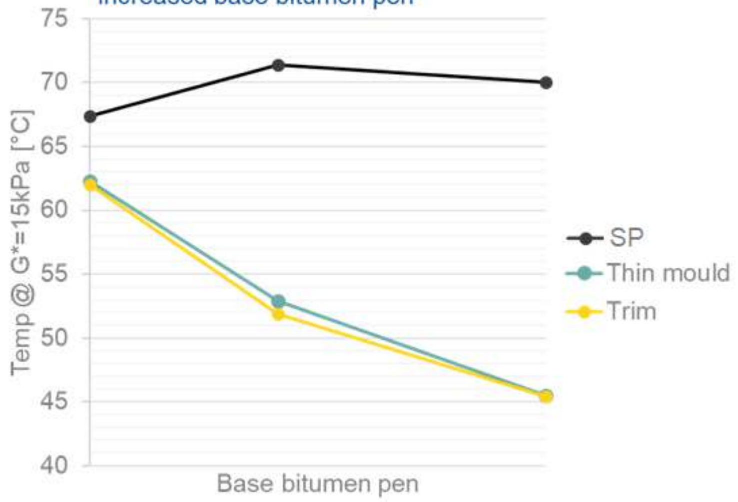
T at G*=15kPa vs softening point

- Not well correlated for PMB

Fixed base binder content with increased polymer content

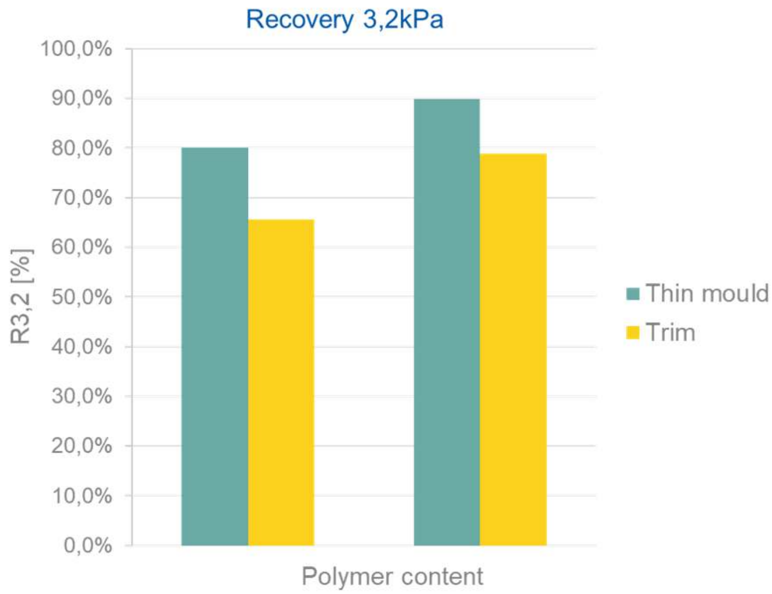


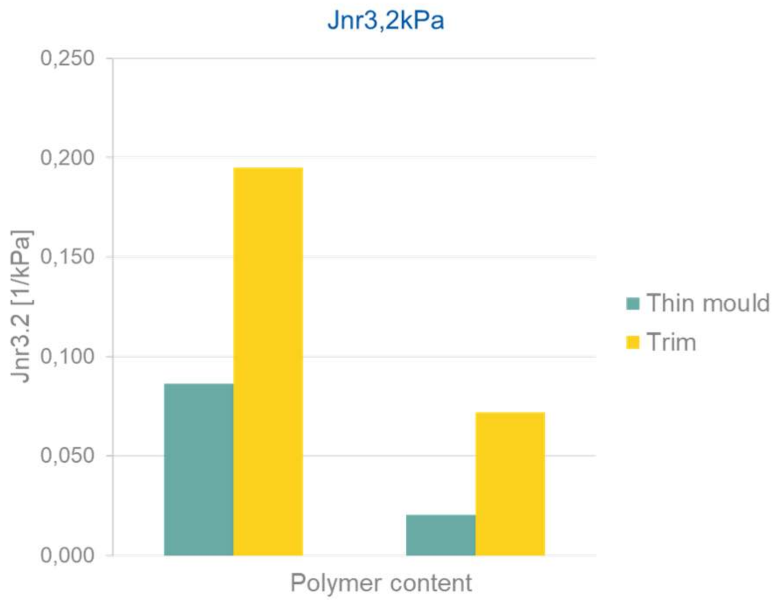
Low fixed polymer content with increased base bitumen pen



MSCR - Thin mould vs trimming

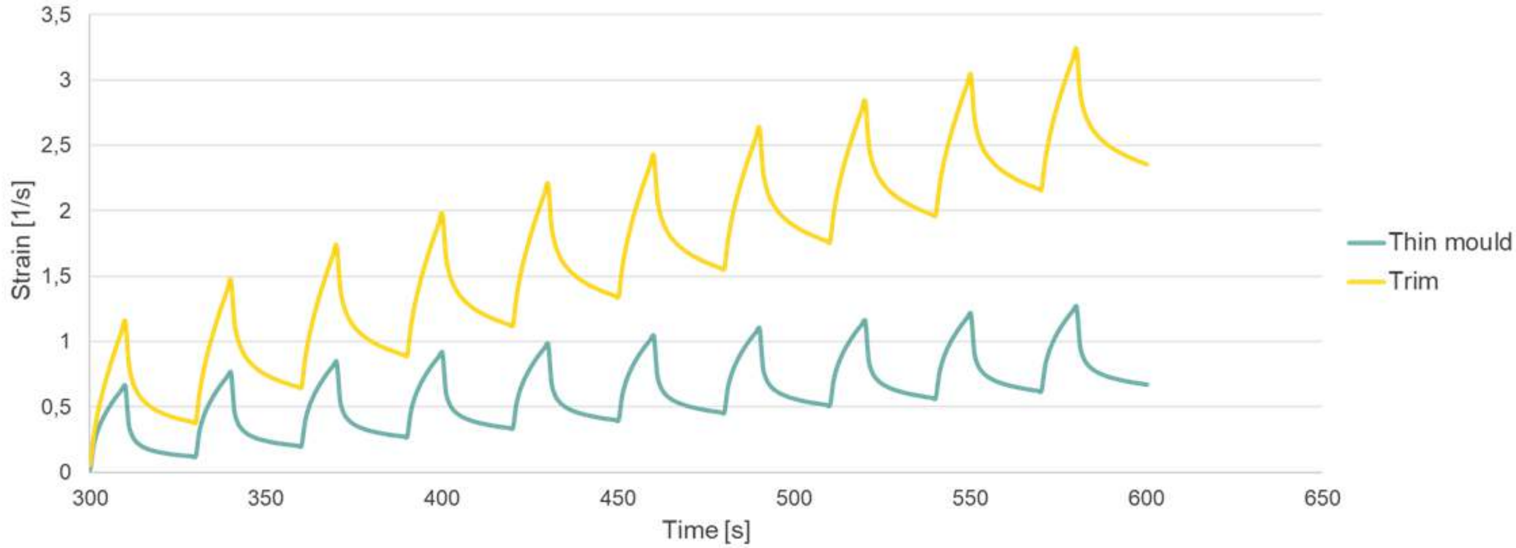
- Fixed base binder with the two higher polymer content PmB





MSCR, Thin mould vs trimming

- Highest polymer content PmB
  - Clear different between sample preparation
  - 3.2 kPa load



DSR sample preparation

- The EN-standard allows a wide interpretation for sample preparation
  - Many laboratories have their own way of preparing the samples
- Not a big problem when penetration grade bitumen is tested;
  But there can be large effects with PmBs,
  - Different results for G*, phase angle (PG grading) and Jnr
  - Effect is larger with higher polymer contents
  - Could explain the differences in DSR-results seen in Round Robins
- DSR is an outstanding device to analyse the binder properties, but for specification purposes, the sample preparation methods need to be standardized

Handbook N200 Vegbygging

(Handbook N200 Road Construction)

- **New requirements for PmBs**
- **Force ductility at 10°C**
  - **Only TBR in previous version**

Tabell 651.3 Krav til polymermodifisert bitumen

	Enhet	Provningsmetod NS-EN	Gradering				
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Flammepunk	°C	ISO 2592	≥220	≥220	≥220	≥220	≥220
Lagringsstabilitet 72 timer ved 180°C		13399	Krav til lagringsstabilitet				
Forsell i mykningspunkt	°C	1427	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
Forsell i penetrasjon	0,1 mm	1426	≤ 9	≤ 9	≤ 9	≤ 9	≤ 9
			Krav til gjenværende egenskaper etter korttidsaldring				
Motstand mot oppherdning, RTFOT ved 163°C		12607-1					
Masseendring	%	12607-1	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5
Gjenværende penetrasjon	%	1426	≥60	≥60	≥60	≥60	≥60
Økning i mykningspunkt	°C	1427	≤ 10	≤ 10	≤ 10	≤ 10	≤ 10
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MSCRT Jnr3,2 kPa ved 60°C	kPa^{-1}	16659	≤ 0,5	≤ 0,2	≤ 1,0	≤ 0,2	≤ 0,1
Temperatursensitivitet T for G*=15 kPa @ 1,59 Hz	°C	14770 med 25 mm plate	≥50	≥55	≥40	≥55	≥60
Temperatursensitivitet T for G*=5000kPa @ 1,59 Hz	°C	14770 med 8 mm plate	≤ 20	≤ 25	≤ 15	≤ 25	≤ 30
RTFOT ved 163°C etterfulgt av PAV ved T=100°C i 20 timer		12607-1 + 14769	Krav til gjenværende egenskaper etter kort- og langtidsaldring				
BBR etter langtidsaldring T (S=300 Mpa)	°C	14771	≤ -15	≤ -15	≤ -21	≤ -24	≤ -12

Experimental

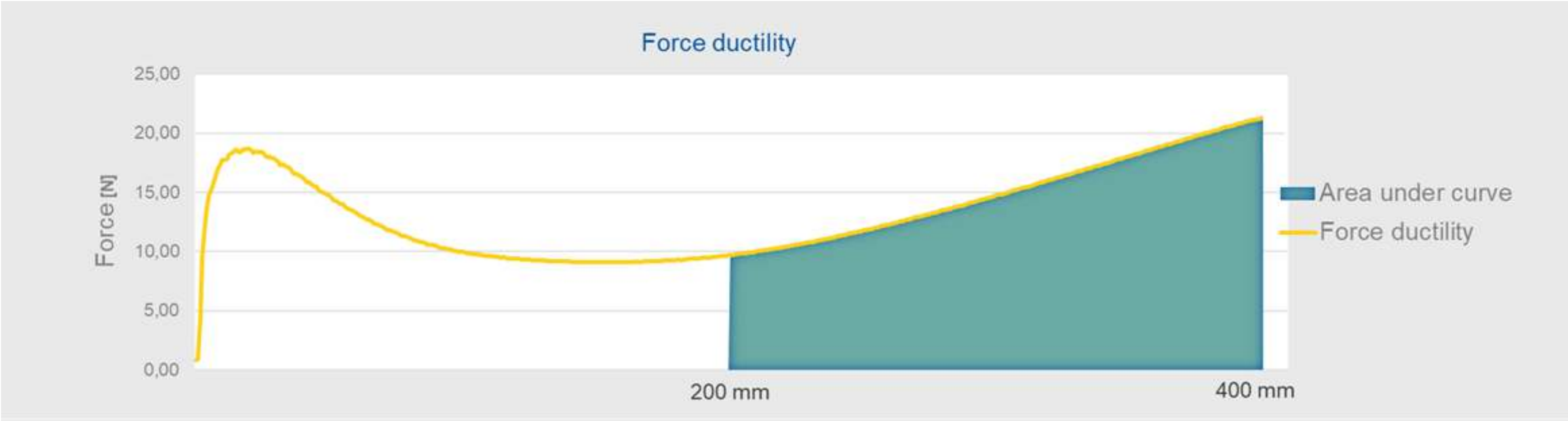
- PMBs with different polymer content and different base binder hardness
- Force ductility at 10°C

Force ductility

- Test cohesion of binder expressed in J/cm^2
- A moulded test specimen is extended in a ductilometer at 50 mm/min
- Usually tested at 5°C but can be increased in steps of 5°C
- Deformation energy is determined from the tensile curve; force versus elongation, by calculating the area under the recorded curve.

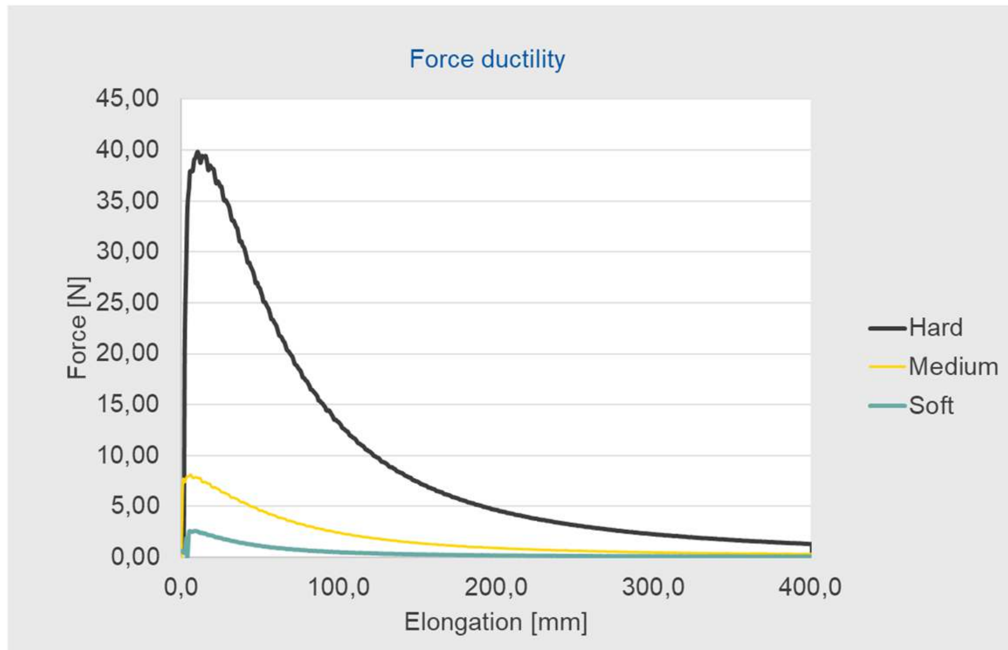
Force ductility

- Deformation Energy = Area under curve
- Cohesion energy is calculated by dividing the deformation energy by the initial cross section of the test specimen
- Force ductility was tested on the same sample as used in the DSR tests



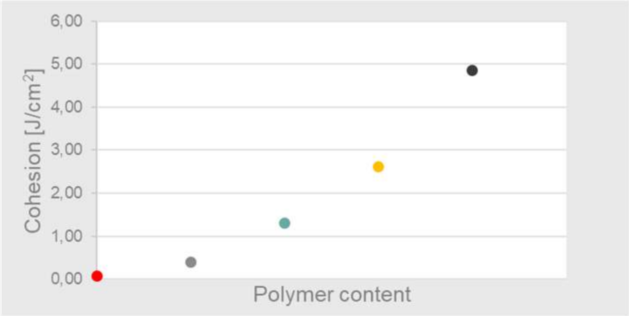
Force ductility

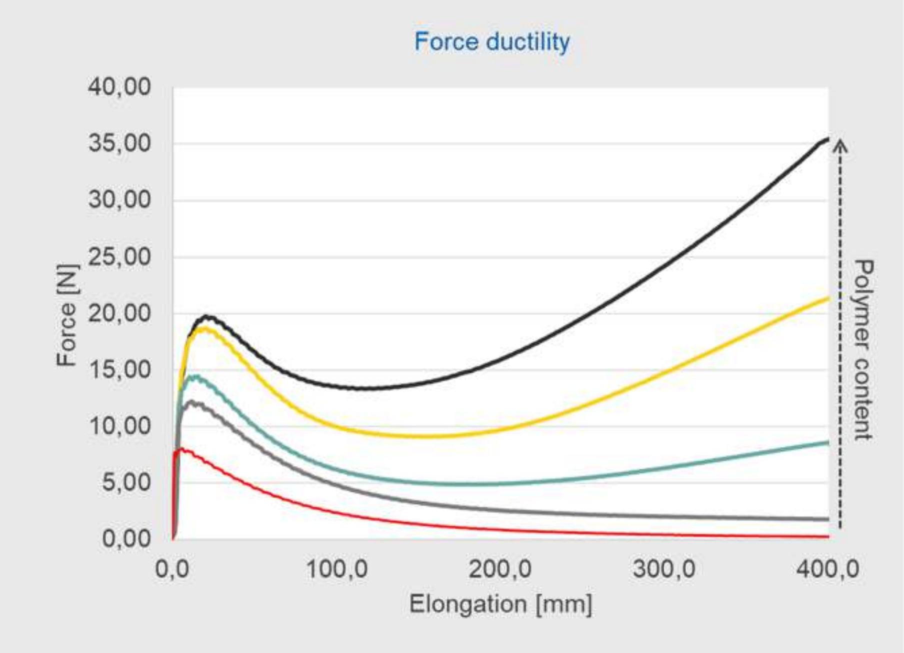
- Three unmodified bitumen tested at 10°C
- Similar shape, different values
- Cohesion energy is dependent on the base binder stiffness
- Largest difference between 0 - 200 mm elongation



Force ductility

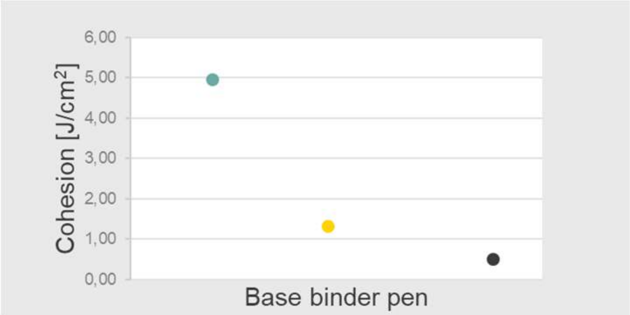
- Fixed base binder with increased polymer content
- Tested at 10°C
- First 50mm is mostly base binder property
- 200 – 400mm is mostly polymer property
- Cohesion correlates to polymer content

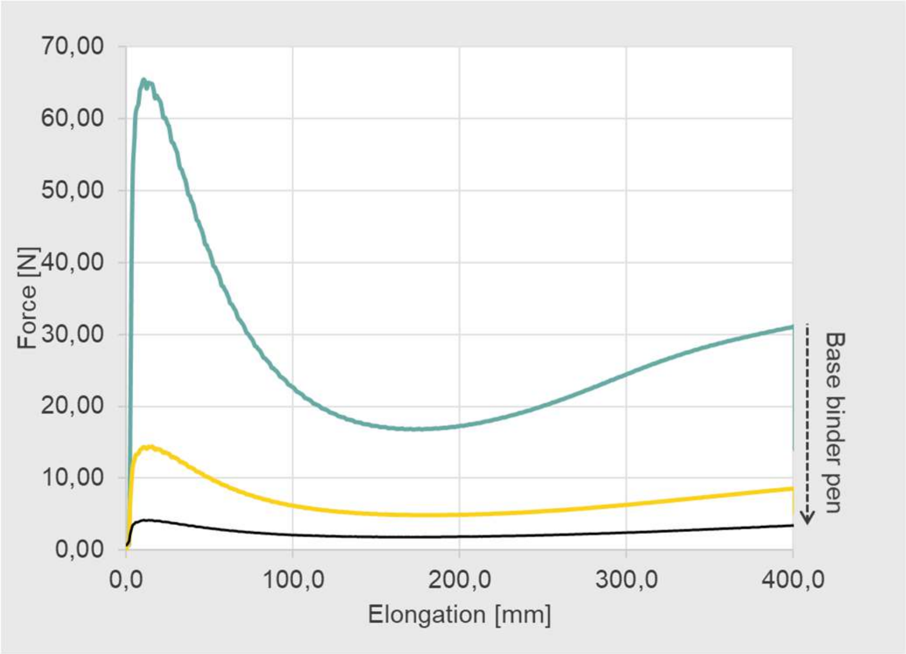




Force ductility

- Low fixed polymer content with increased base bitumen pen
- Tested at 10°C
- Pen of base bitumen has a big impact on cohesion energy





Cohesion by force ductility

- Cohesion energy is effected by
  - base binder pen
  - polymer content

- Different ways to achieve high cohesion (high polymer content or harder base binder)
  - Both ways will decrease the penetration of the PmB
  - A PmB with a high cohesion has a low(er) penetration

- Combinations of both high penetration and high cohesion is hard to achieve
  - Like 75/130-80 which should have a cohesion >2 J/cm^2 at 10° in N200

Summary

DSR

- Outstanding device to analyse the binder properties and has good potential
- The DSR EN-14770 standard allows a wide variation of sample preparation methods
- PMB morphology / polymer network is affected
- When using DSR values for specification limits, preparation methods need to be better defined

Force Ductility

- Cohesion energy is affected by penetration of base binder and polymer content
- The combination of a high penetration and a high cohesion energy is hard to achieve

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