Novel Cured Silicone And Silicone/Organic Hybrid Systems And Their Properties: Epoxies

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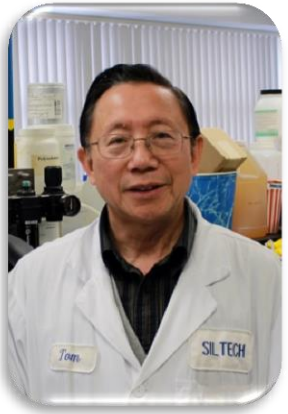

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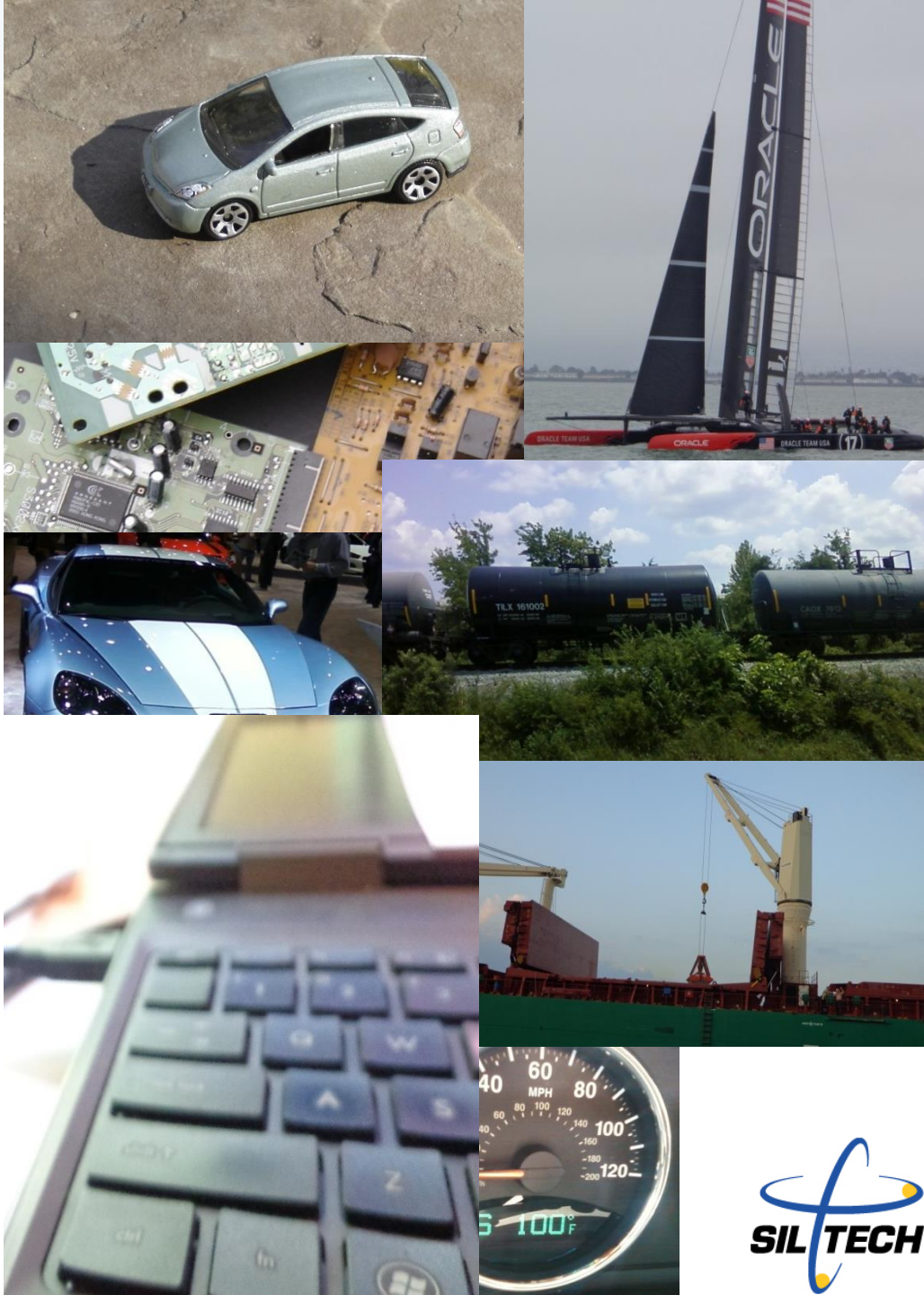
Epoxy Resins

- Myriad Applications
  - Adhesives
  - Aerospace
  - Coatings
  - Composites
  - Construction
  - Electronics
  - Specialty Applications
  - Transportation



Epoxy Resins

- Thermoset
- Often 2k
- Diverse Base Resins
- Cure Mechanisms
  - Amine
  - Mercaptan
  - Anhydride
  - UV Initiated Acid
- Modifiers

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Epoxy Resin Properies

- Few Compromises
- Solvent Resistance
- Low Shrinkage
- Processability
- Insulative
- Adhesion
- Strength
- Relatively Brittle

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Need for Flexible Epoxies

- Adhesives
- Composites
- Electronics
- Floors
- Marine
- Plastics
- Wood

Silicones

- Incompatible
- Gas Permeable
- Low Tg: -120°C
- Low Order of Toxicity
- Flexible, Compressible
- Low Surface Energy (ST, COF)
- Very Good Thermal Flexibility
- Excellent Spreading and Wetting
- Insulative (Electrical and Thermal)
- Thermal and Radical Stable (O_2, O_3, Sunlight)
- Good Chemical and Very Good Water Resistance

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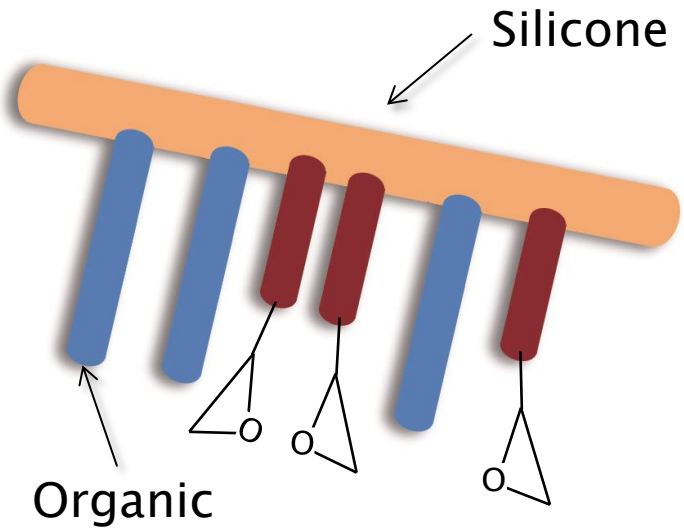
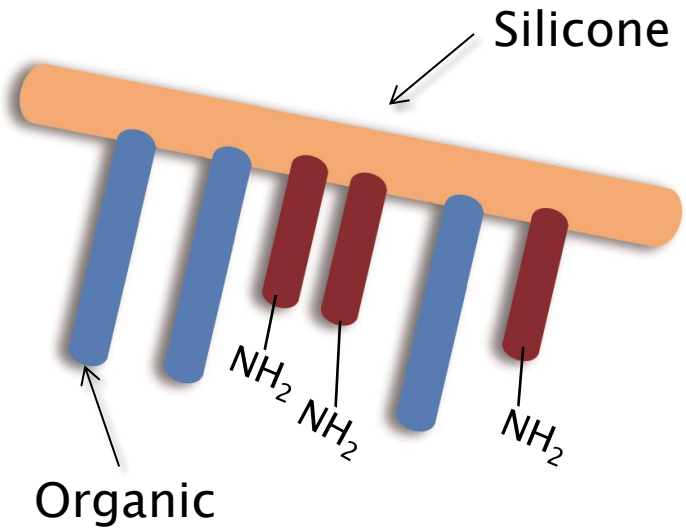
Reactive Silicones

- Silicones can by synthesized with a variety of reactive groups including cycloaliphatic or glycidyl epoxy moieties.
- These can be reacted as homopolymers or copolymers with other epoxy resins



Reactive Silicones as Hardeners

- …or amine functionality
- These reactive silicones can be used as “flexible hardeners.”
- The organic groups provide solubility.



Silicones Used

Silicone	# Reactive Sites	Equivalent Weight	Organic Group
Epoxy A	1 EP/3 OH	2400	Polyether
Epoxy B	1 EP/5 OH	8200	Polyether
Hydroxyl A	3 OH	3800	None
Hydroxyl B	2 OH	1980	None
Hydroxyl C	4 OH	360	None
Amine A	4 NH_2	300	None
Amine B	1 NH_2/3 OH	2550	Polyether
Amine C	2 NH_2	450	None

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Epoxy Silicones with Epoxy A

- Dow DER 671-X75, a commercial low MW, Epichlorohydrin/ bisphenol A system is reacted with epoxy silicone A.
- MHHPA and Imicure AM-1used to affect cure.
- Cured at 100°C for 4 hours.
- Properties are followed with Brookfield DV-III Rheometer AR-G2 or measured with Instron #1122.

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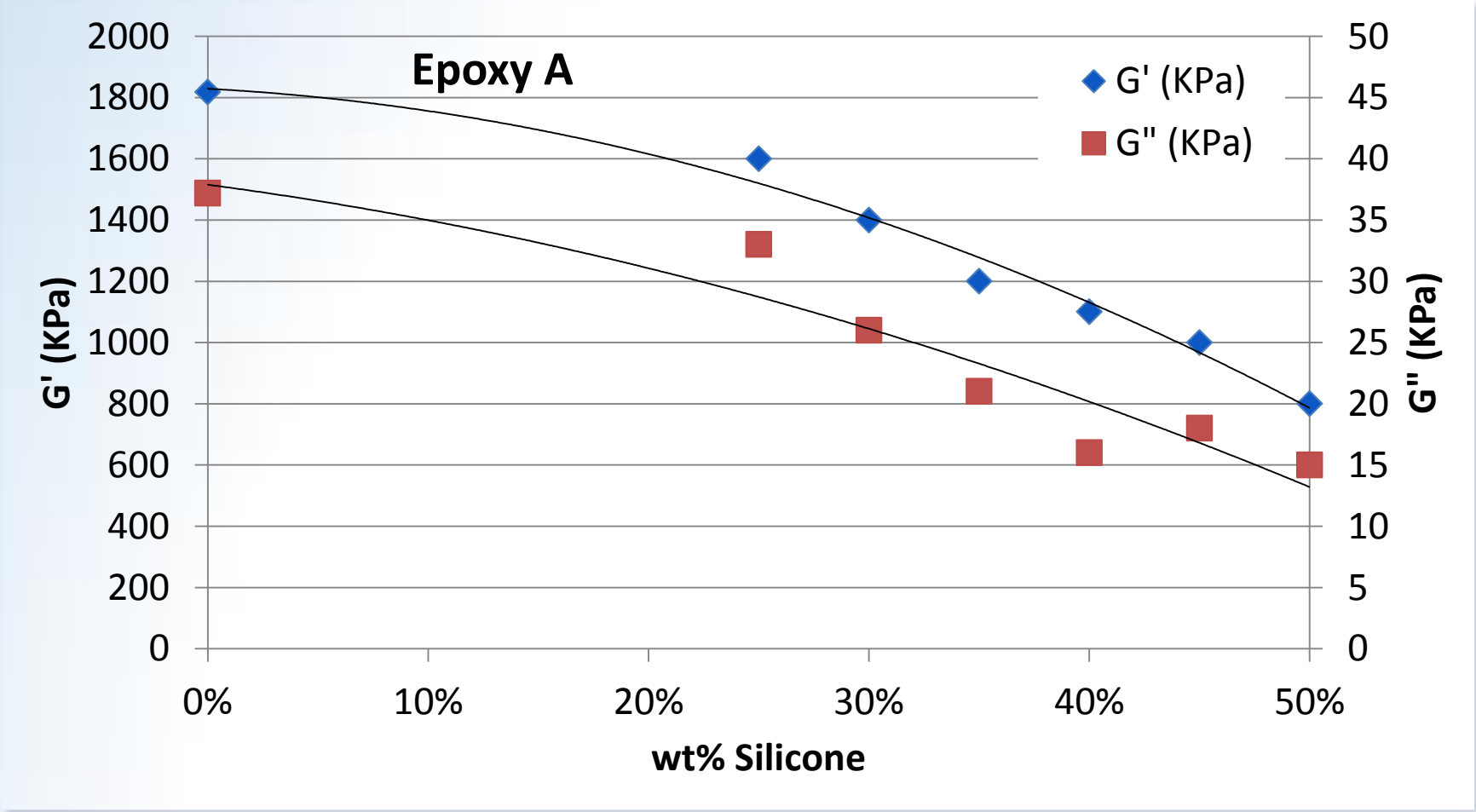
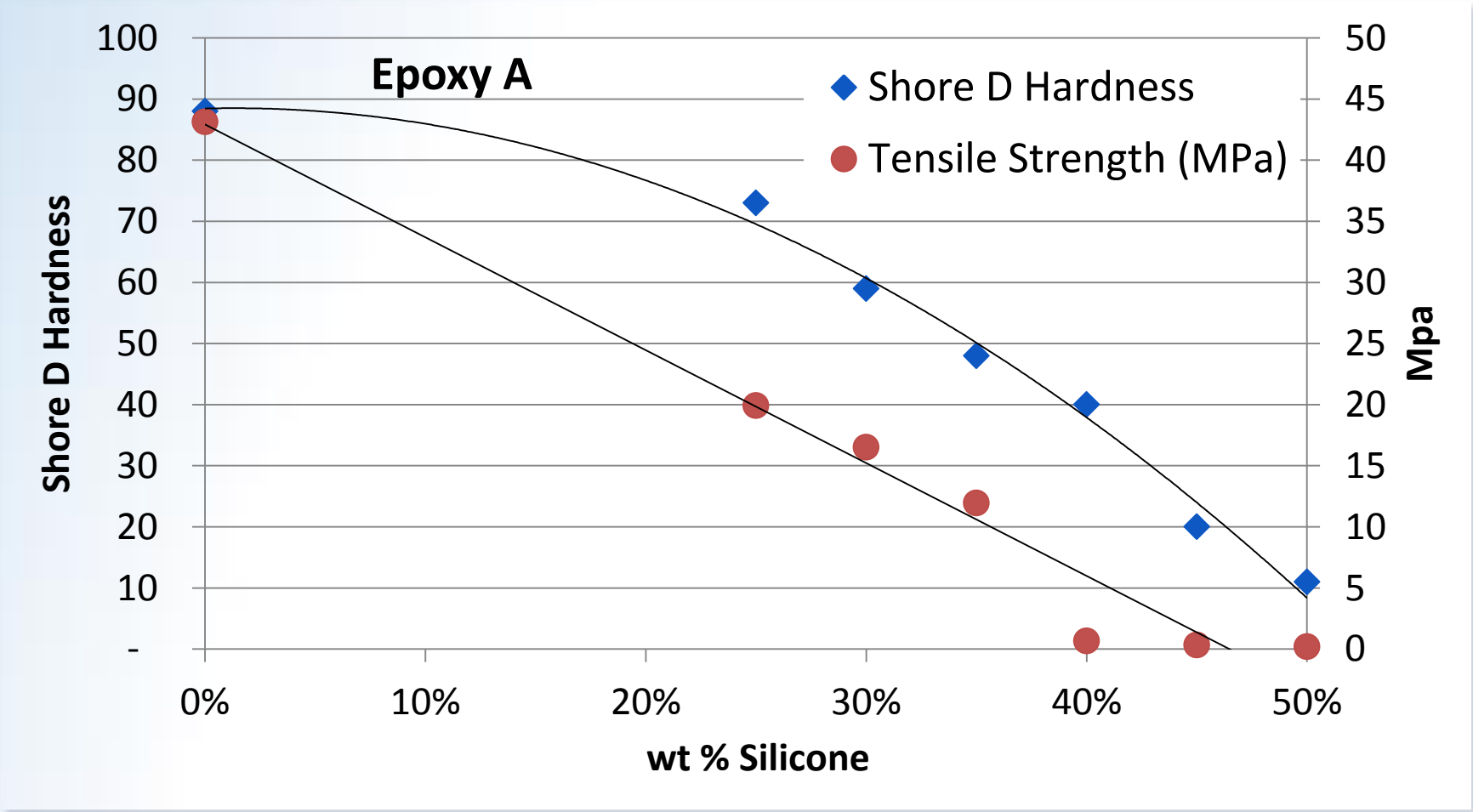
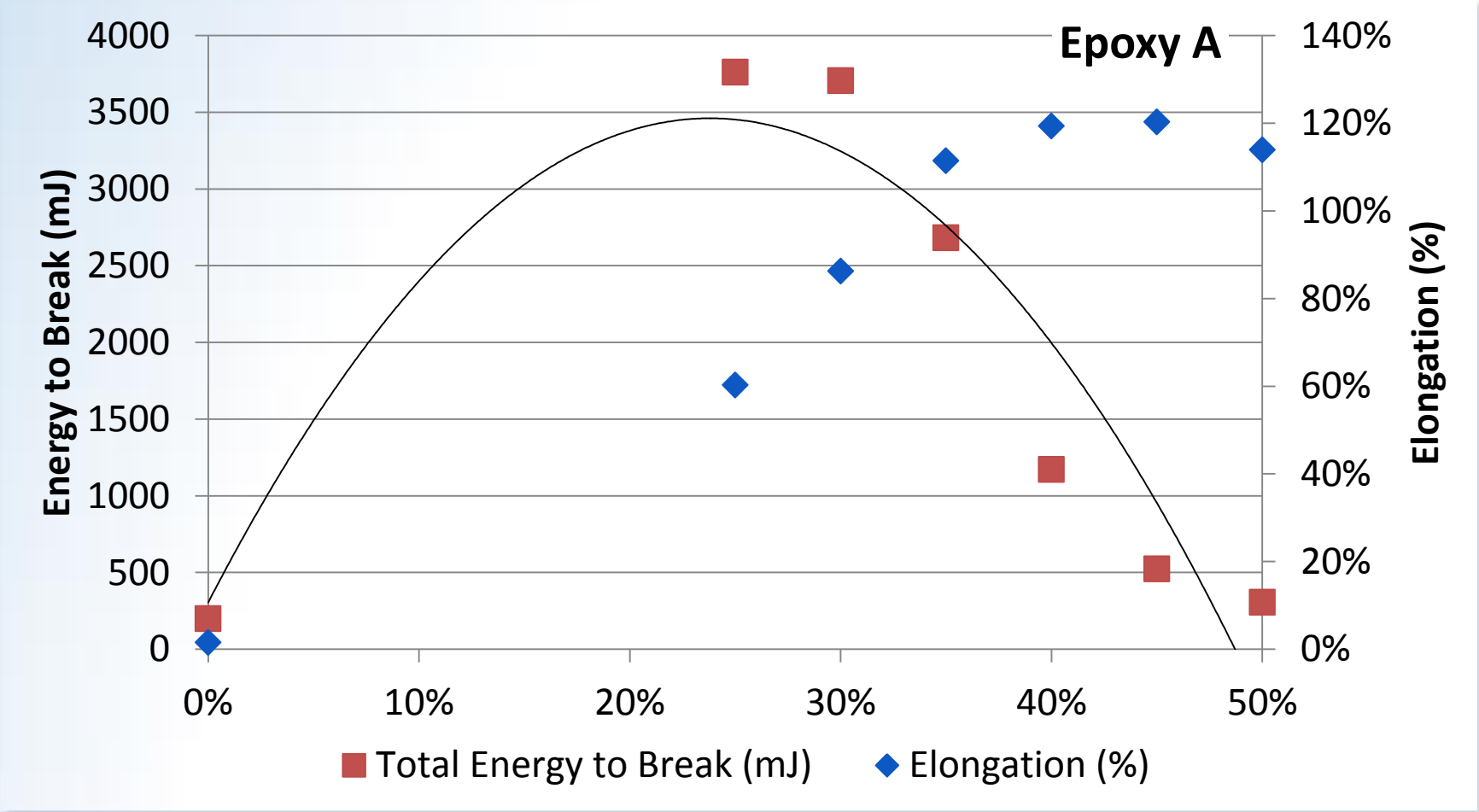
Modulii


Epoxy A
G' (KPa)
G" (KPa)
2000
1800
1600
1400
1200
1000
800
600
400
200
0
50
45
40
35
30
25
20
15
10
5
0
0%
10%
20%
30%
40%
50%
wt% Silicone

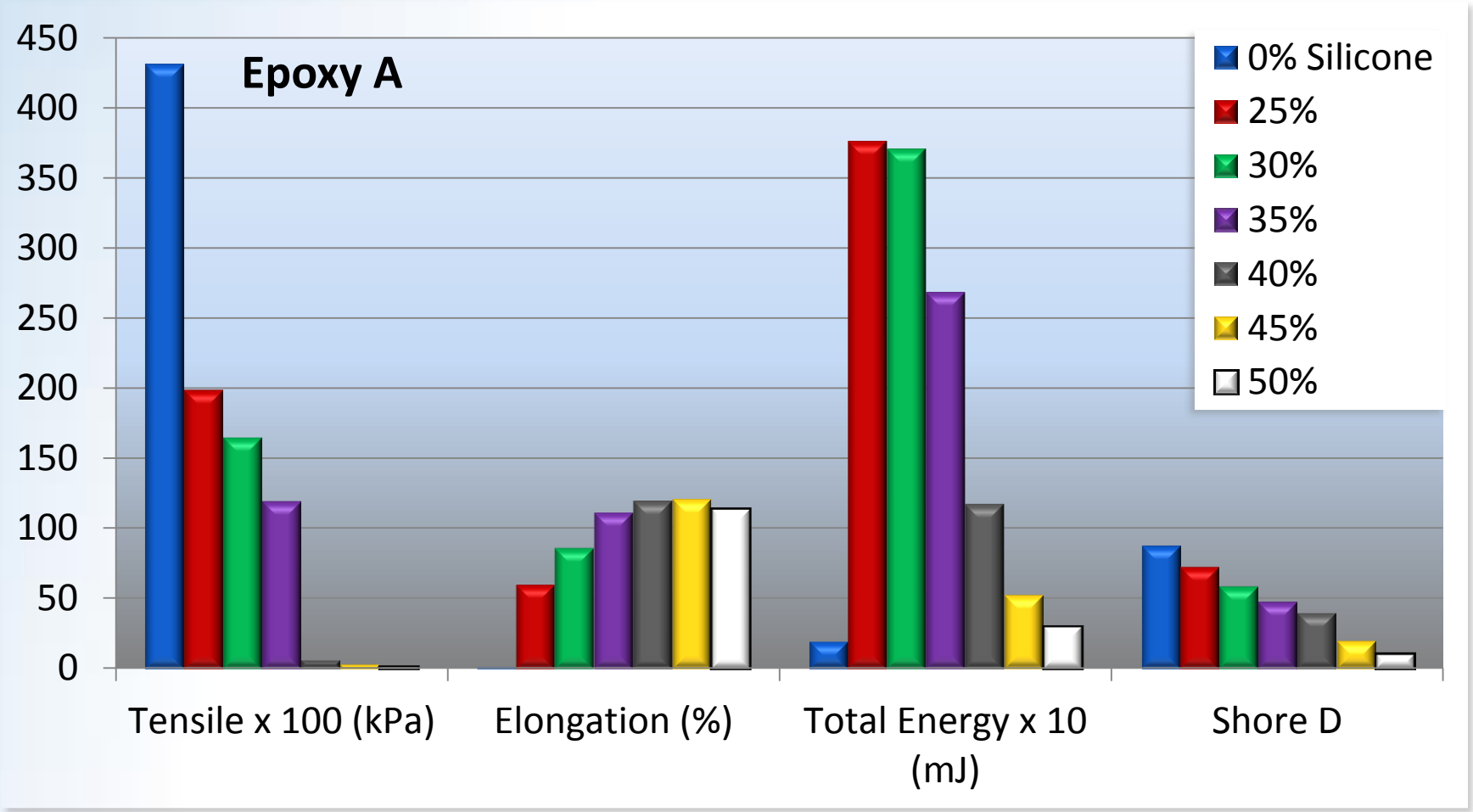
Hardness and Strength



Total Energy to Break/ Elongation



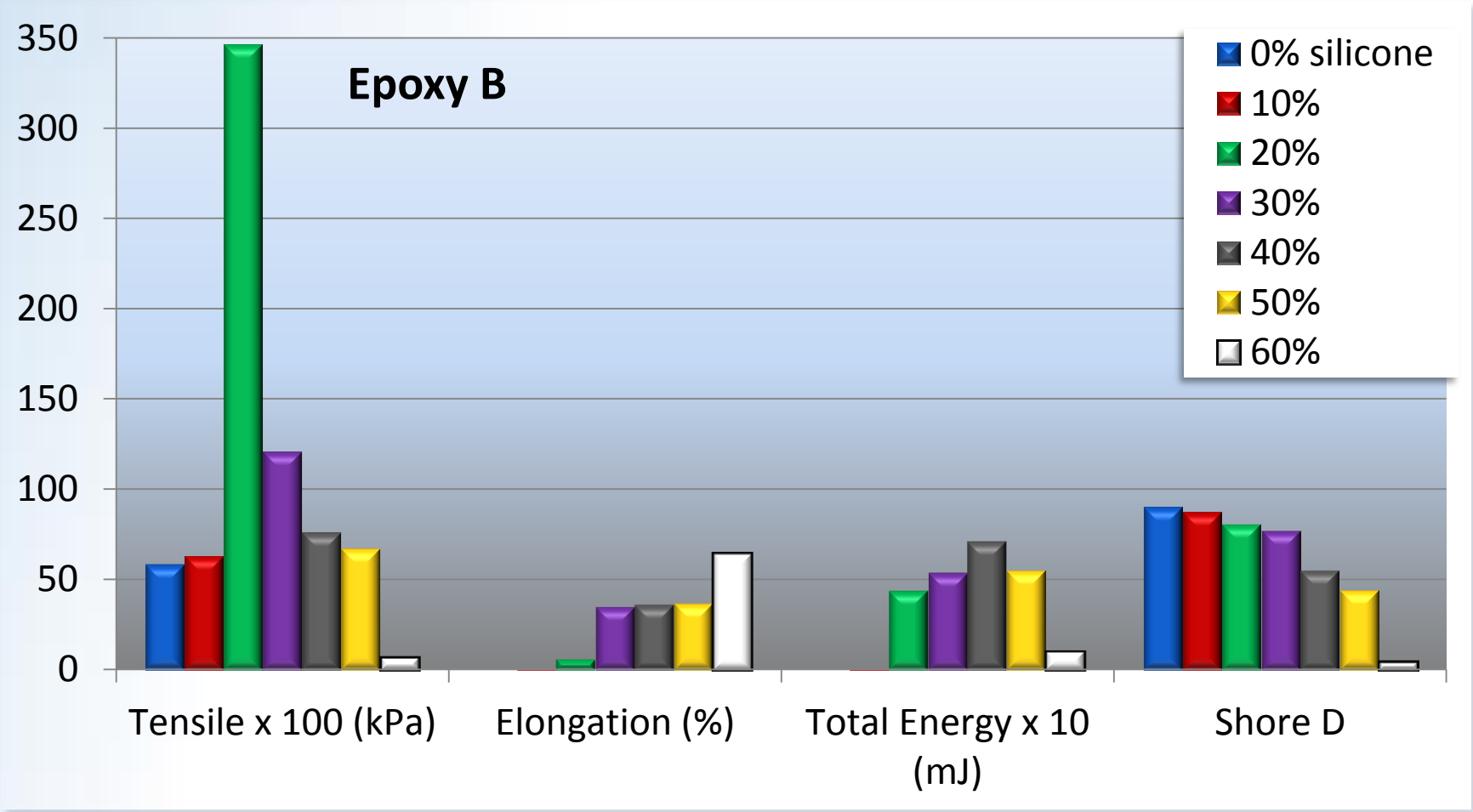
Properties Epoxy A



Epoxy Silicones with Epoxy B

- UVACure 1500, a commercial cycloaliphatic epoxy system is reacted with epoxy silicone B
- MHHPA and Imicure AM-1used to affect cure.
- Cured at 100°C for 4 hours.
- Properties are followed with Brookfield DV-III Rheometer AR-G2 or measured with Instron #1122.

Properties with Epoxy B



Different Organic and Silicone Resins

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Amine Hardened Epoxy/ Silicones

- Dow DER 331 Epichlorohydrin/ bisphenol A commercial system is reacted with reactive silicones
- MHHPA and Imicure AM-1used to affect cure.
- Cured at ambient for 24 hours.
- Ancamine 1618 is found to be best for hardening over Ancamine 1483 or TEPA.
- Properties are followed with Brookfield DV-III Rheometer AR-G2 or measured with Instron #1122

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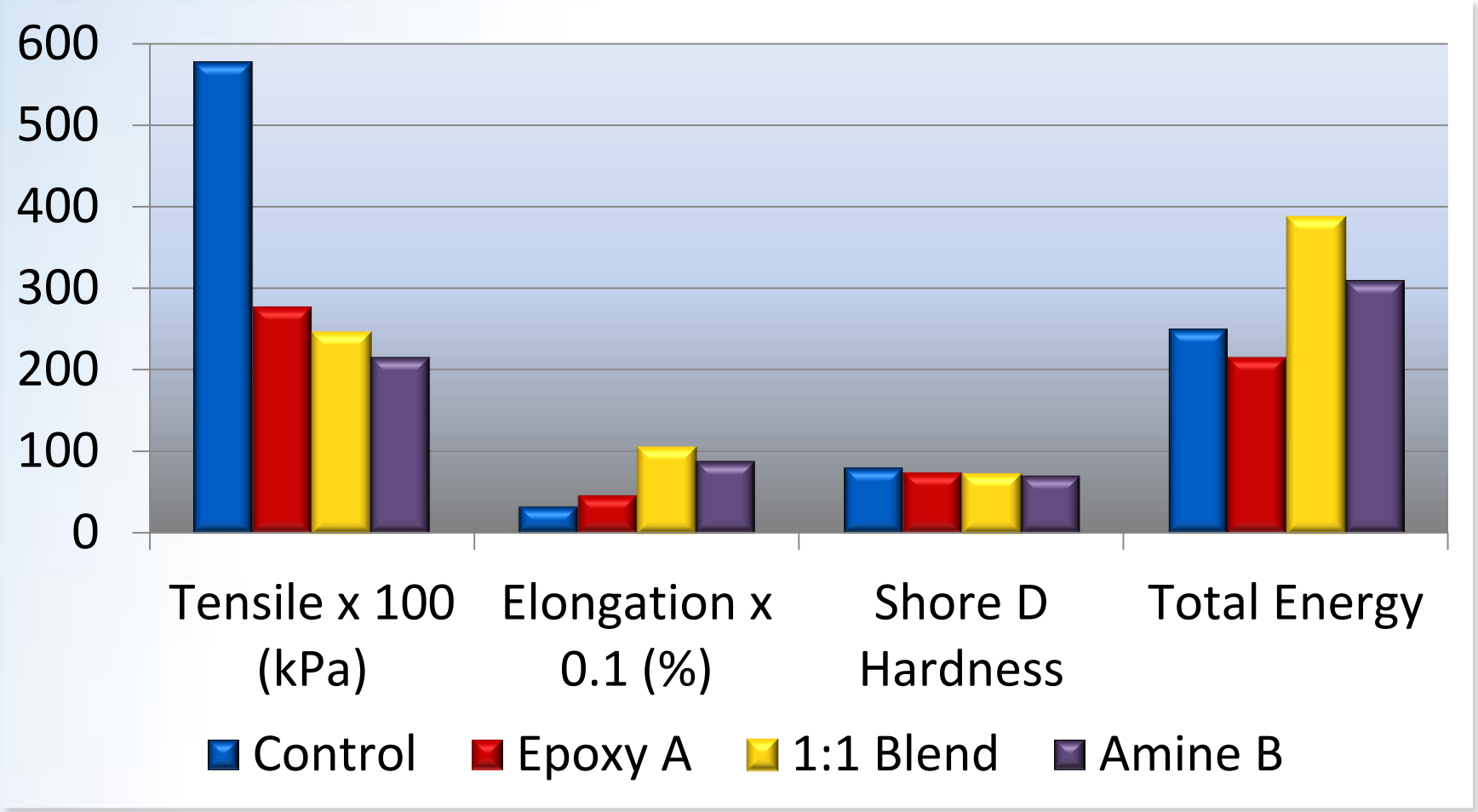
Silicone Hardeners

Replace 20% of Ancamine 1618 (hardener):

- Silicone Amine B
- Silicone Epoxy A
- 1:1 blend of Amine B/ Epoxy A

Results


600
500
400
300
200
100
0
Tensile x 100 (kPa)
Elongation x 0.1 (%)
Shore D Hardness
Total Energy
Control
Epoxy A
1:1 Blend
Amine B

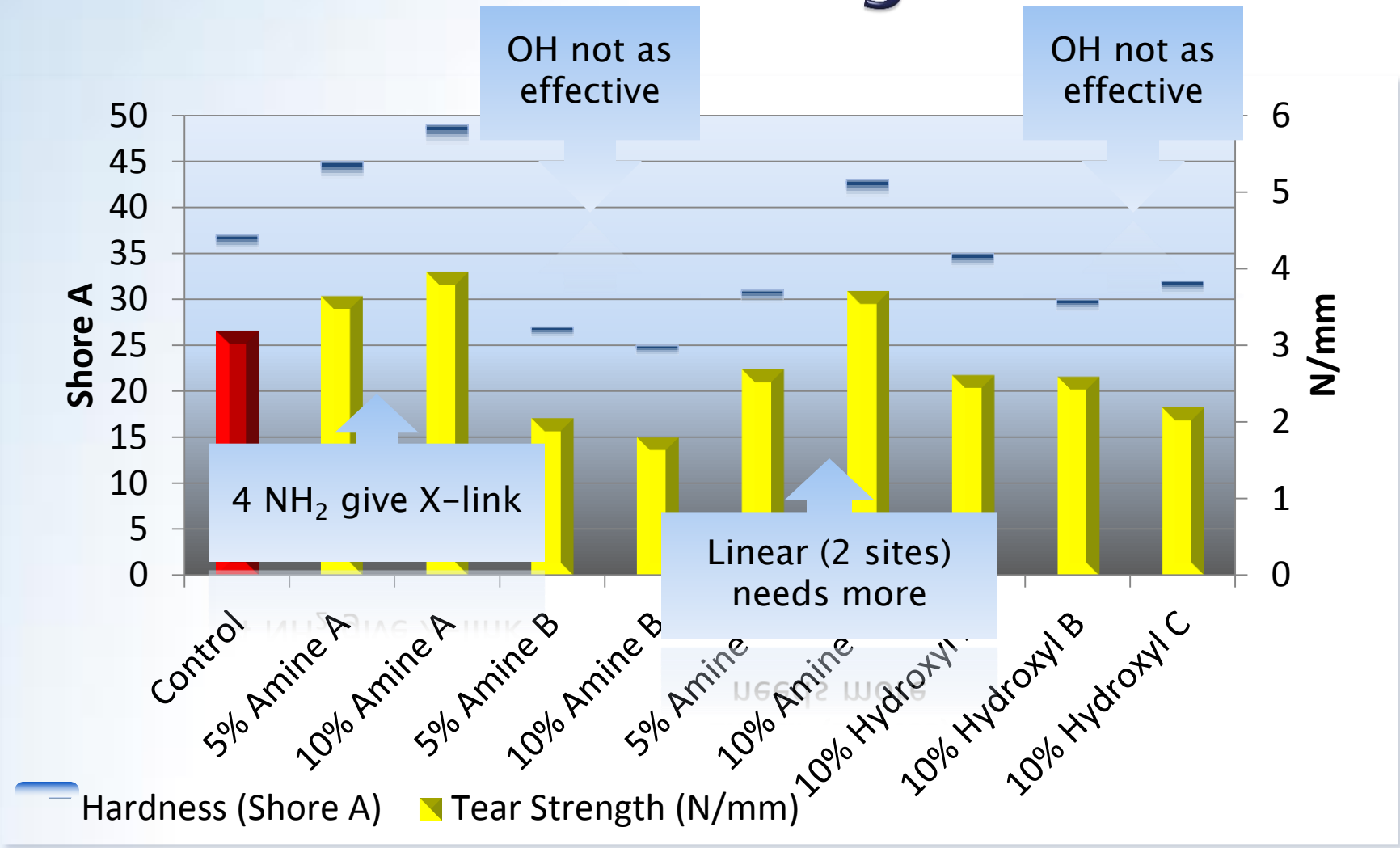
Rubber Filled Epoxy with Silicone Hardener

- Proprietary epoxy with 5-10% reactive silicones and rubber crumbs
- Mold and cure at ambient for 7 days
- -15°C and -30°C impact resistance
- Severity of fracture rated 1-10 (best)

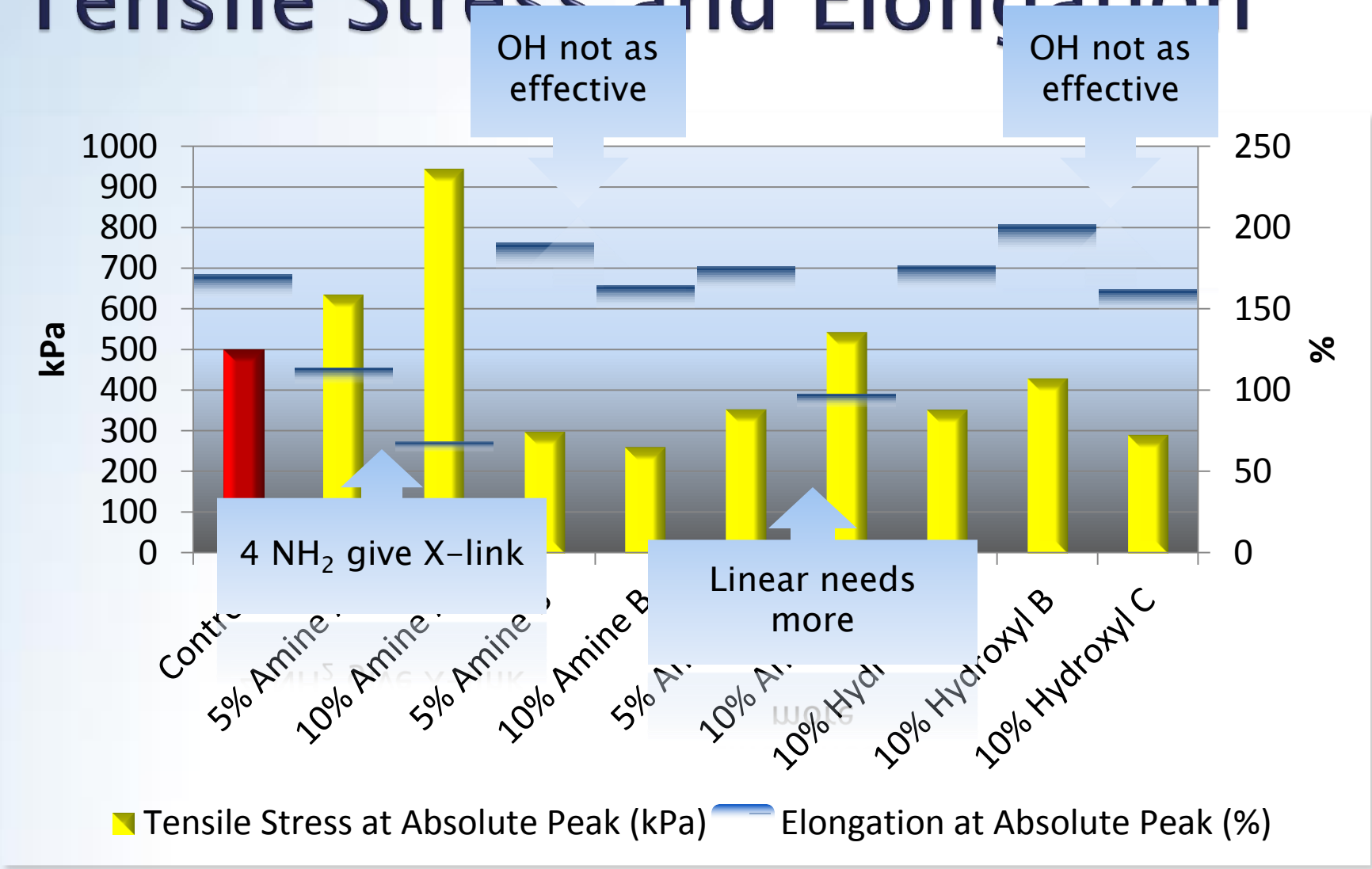
Results

Additive	%	Shore A	Tear (N/mm)	Tensile (kPa)	Elongation (%)	- 15° C	- 30° C
Control	0	37	3.2	500	171	5	6*
Amine A	5%	45	3.6	636	114	8	7*
Amine A	10%	49	4.0	943	68	9.5	9*
Amine B	5%	27	2.1	299	191	5	5.5
Amine B	10%	25	1.8	261	164	4.5	2.5
Amine C	5%	31	2.7	354	176	4.5	3
Amine C	10%	43	3.7	543	98	9.5	8*
Hydroxyl A	10%	35	2.6	353	176	6	5
Hydroxyl B	10%	30	2.6	430	202	3	4
Hydroxyl C	10%	32	2.2	291	162	4	8.5*

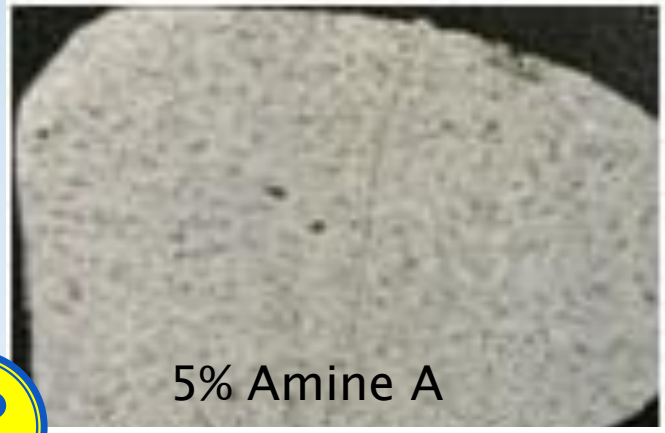
Hardness and Strength

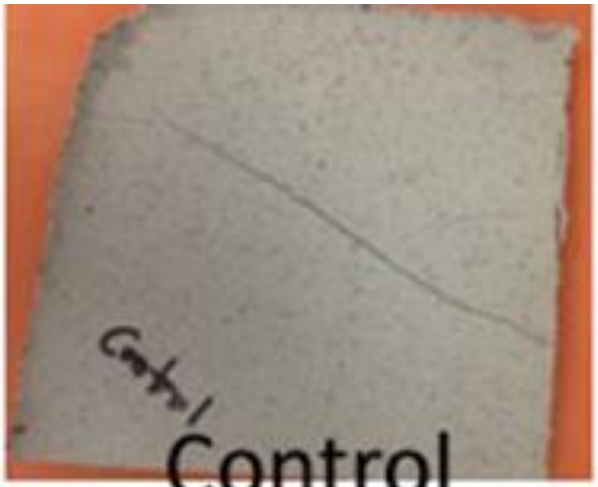


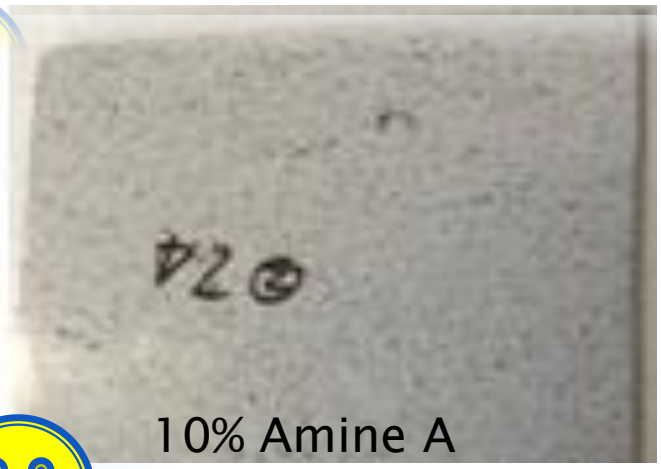
Tensile Stress and Elongation



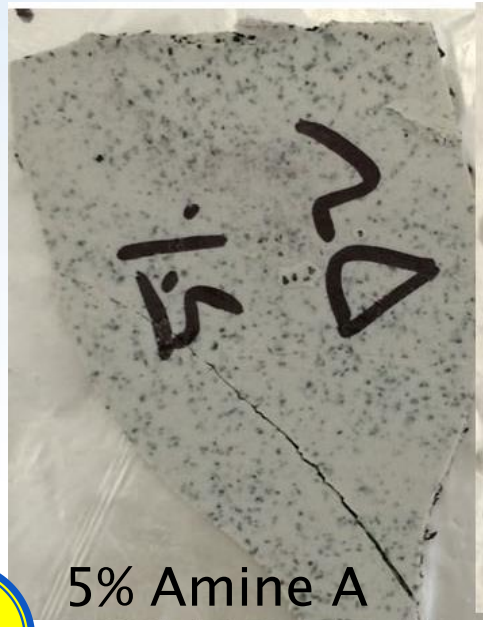
-15°C
Fracture



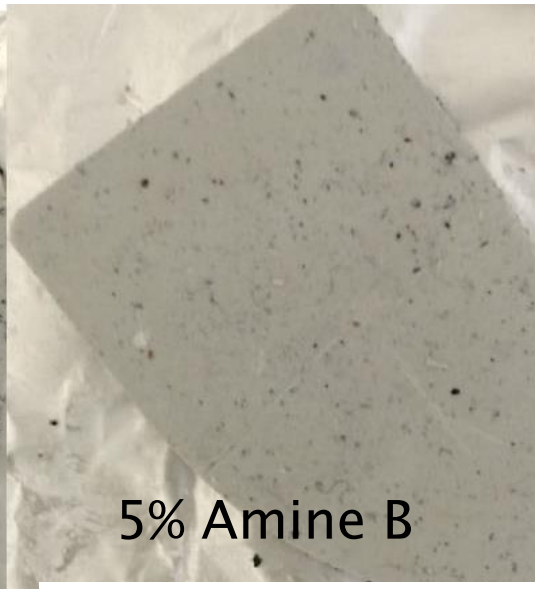




Control

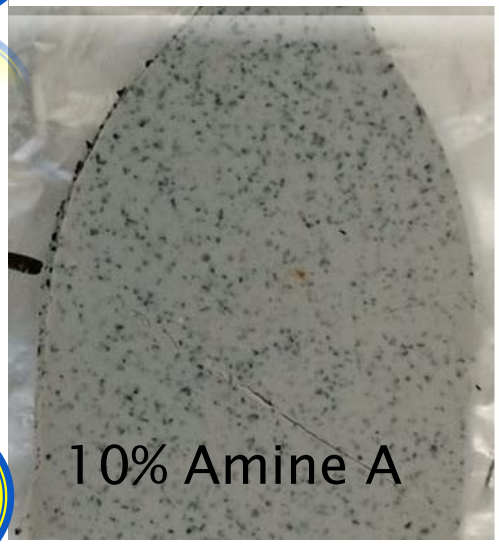


5% Amine A

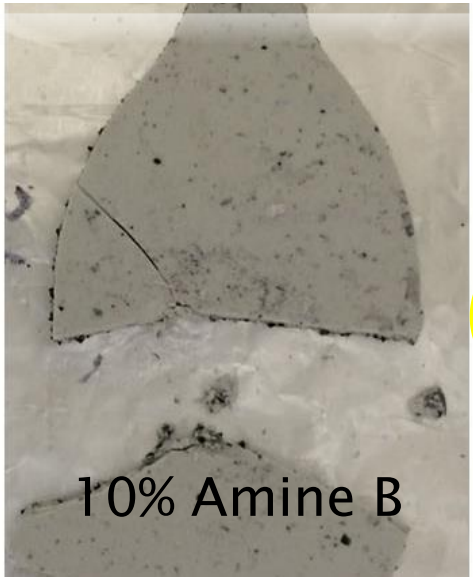


5% Amine B

-30°C
Fracture



10% Amine A



10% Amine B

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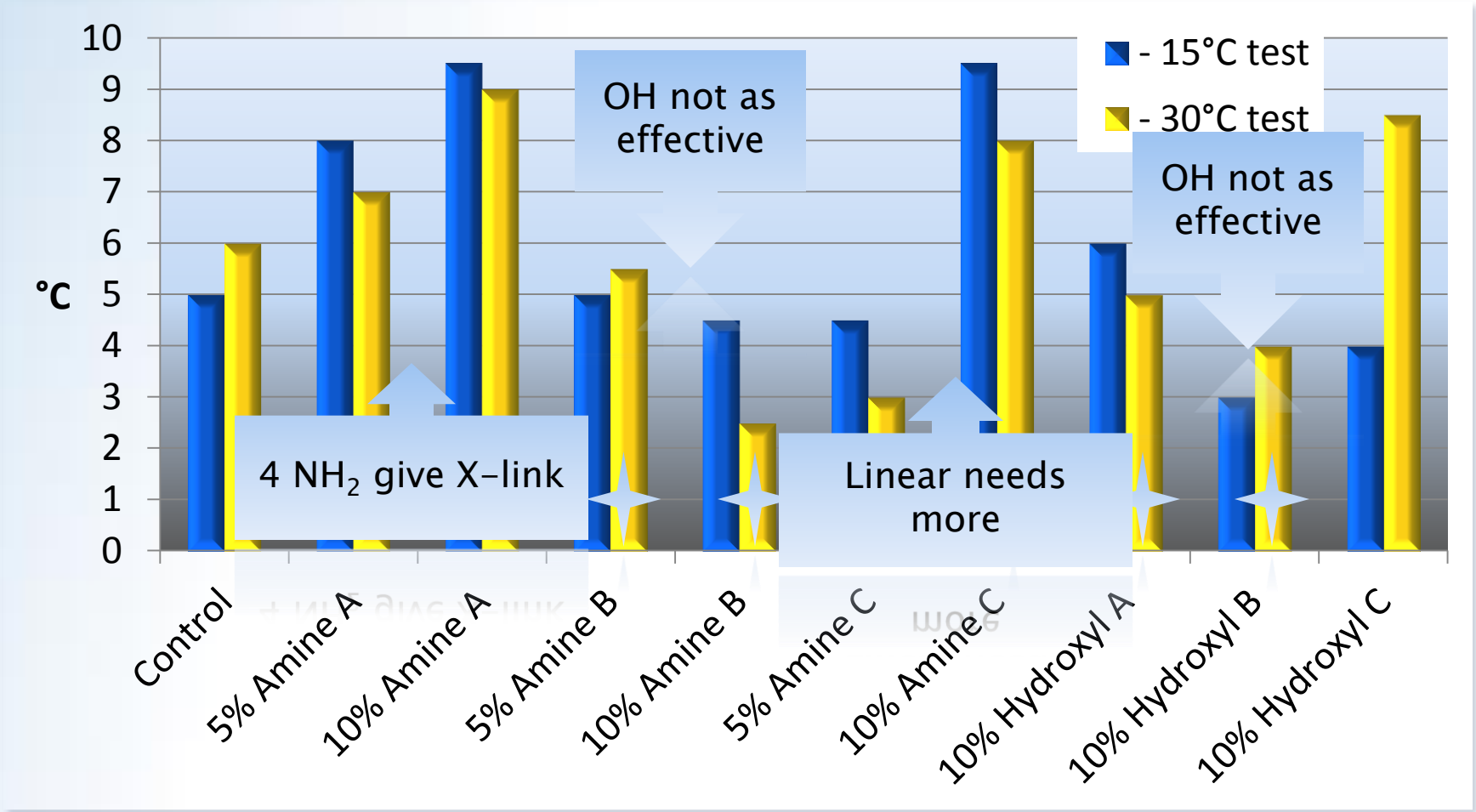
Low Temperature Impact



Fractured on first of two impacts

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Conclusions

- In the Shore D systems, silicone reduces hardness.
  - Slowly up to 20%
- Strength and elongation improve and maximize at ~10-20% silicone.
- Amine more effective than epoxy
- In the Shore A system, with tetra-functional Amine A, hardness is increased.
- Impact resistance is also increased.
- OH is not as effective for this.

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