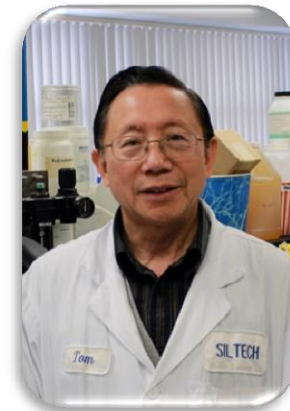


# Novel Cured Silicone And Silicone/Organic Hybrid Systems And Their Properties: Epoxies

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Siltech Corporation



# 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



# Epoxy Resin Properties

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



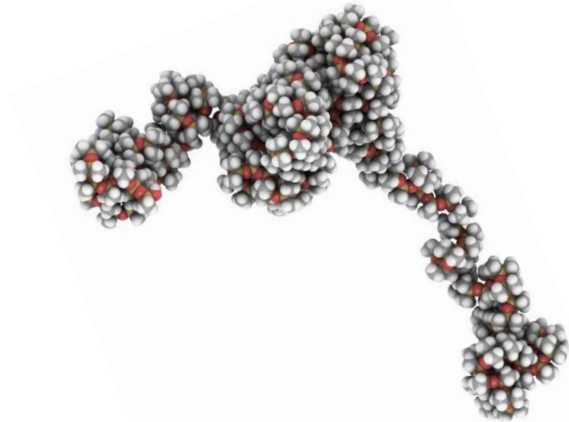
# Need for Flexible Epoxies

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



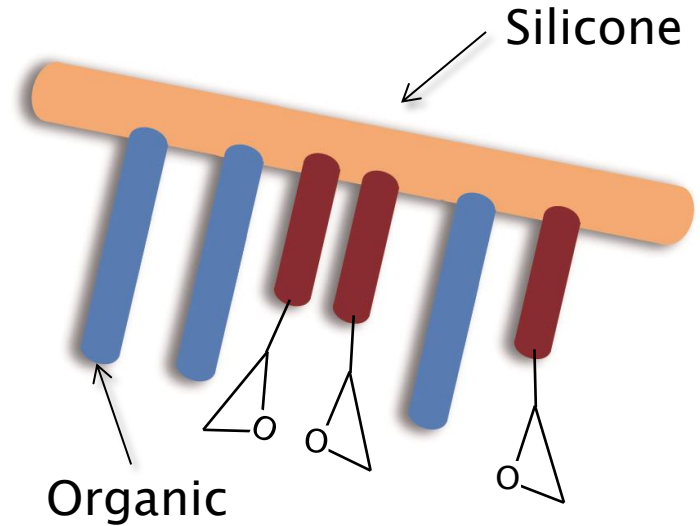
# Silicones

- ▶ Incompatible
- ▶ Gas Permeable
- ▶ Low Tg:  $-120^{\circ}\text{C}$
- ▶ 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 ( $\text{O}_2$ ,  $\text{O}_3$ , Sunlight)
- ▶ Good Chemical and Very Good Water Resistance



# Reactive Silicones

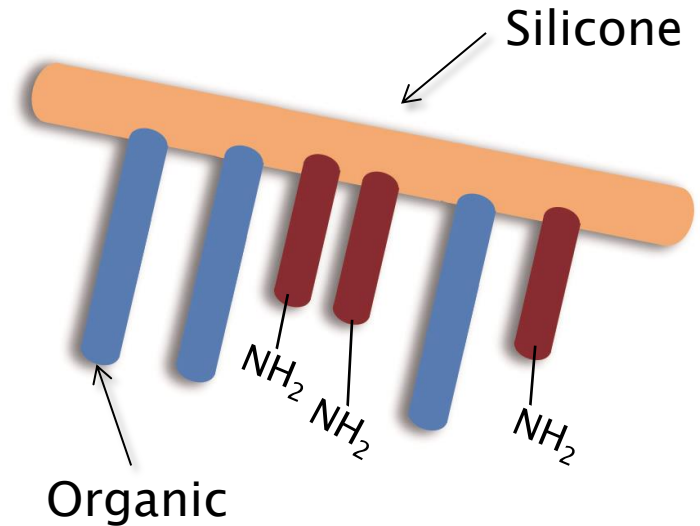
- ▶ Silicones can be synthesized with a variety of reactive groups including cycloaliphatic or glycidyl epoxy moieties.
- ▶ These can be reacted as homopolymers or copolymers with other epoxy resins





# Reactive Silicones as Hardeners

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





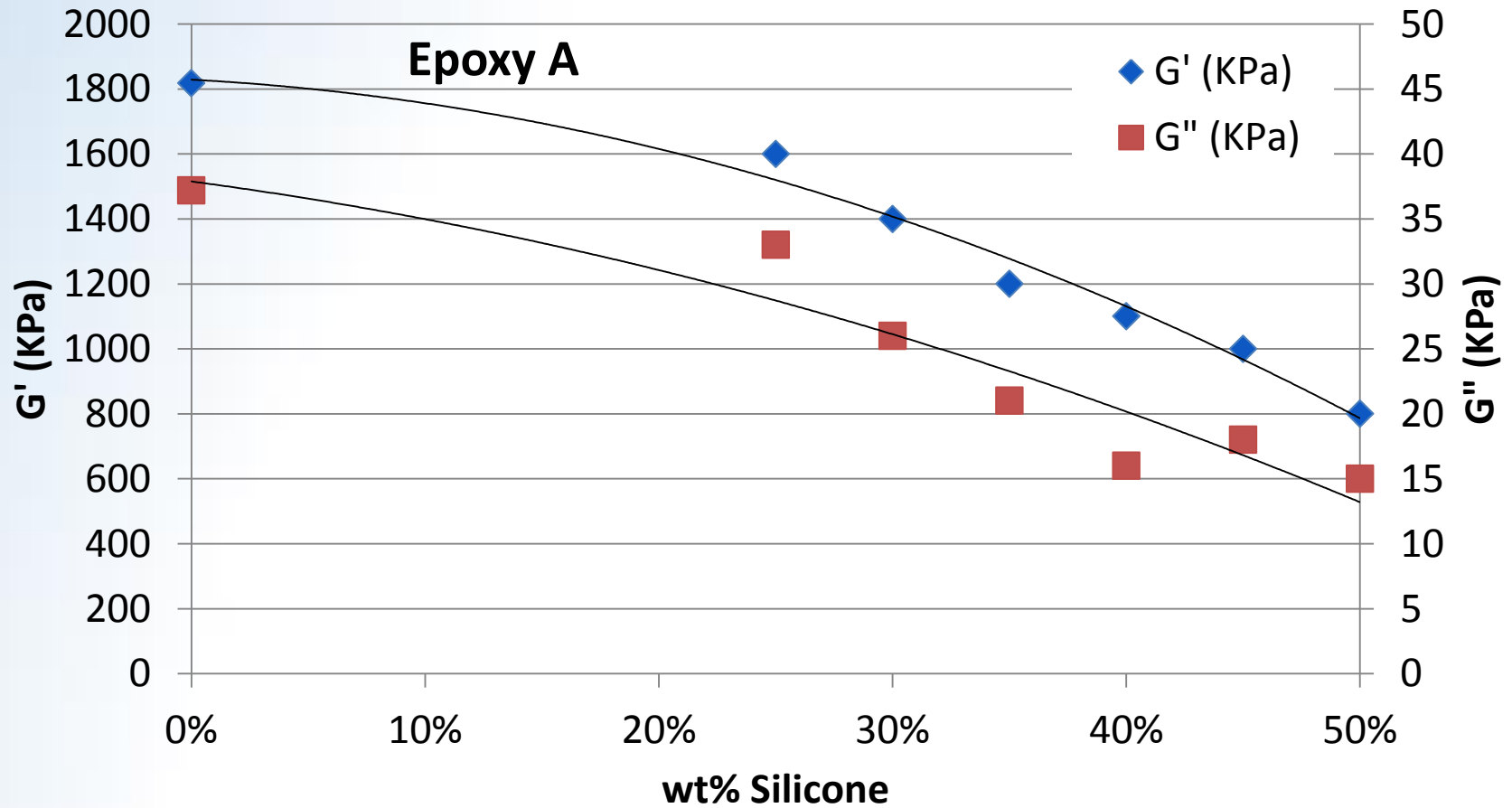
# Silicones Used

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 <sub>2</sub>	300	None
Amine B	1 NH <sub>2</sub> /3 OH	2550	Polyether
Amine C	2 NH <sub>2</sub>	450	None

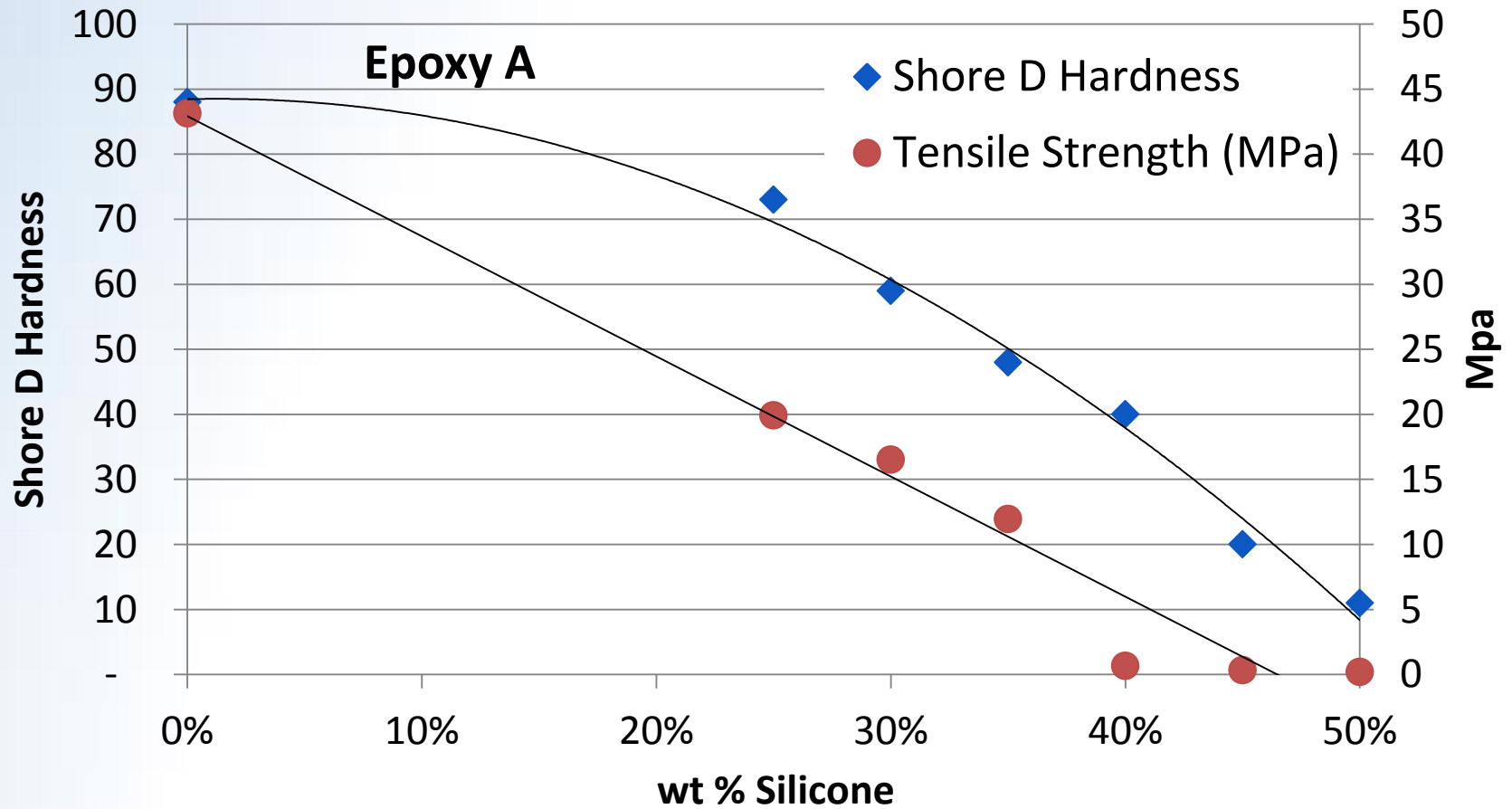
# Epoxy Silicones with Epoxy A

- ▶ Dow DER 671-X75, a commercial low MW, Epichlorohydrin/ bisphenol A system is reacted with epoxy silicone A.
- ▶ MHHPA and Imicure AM-1 used 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.

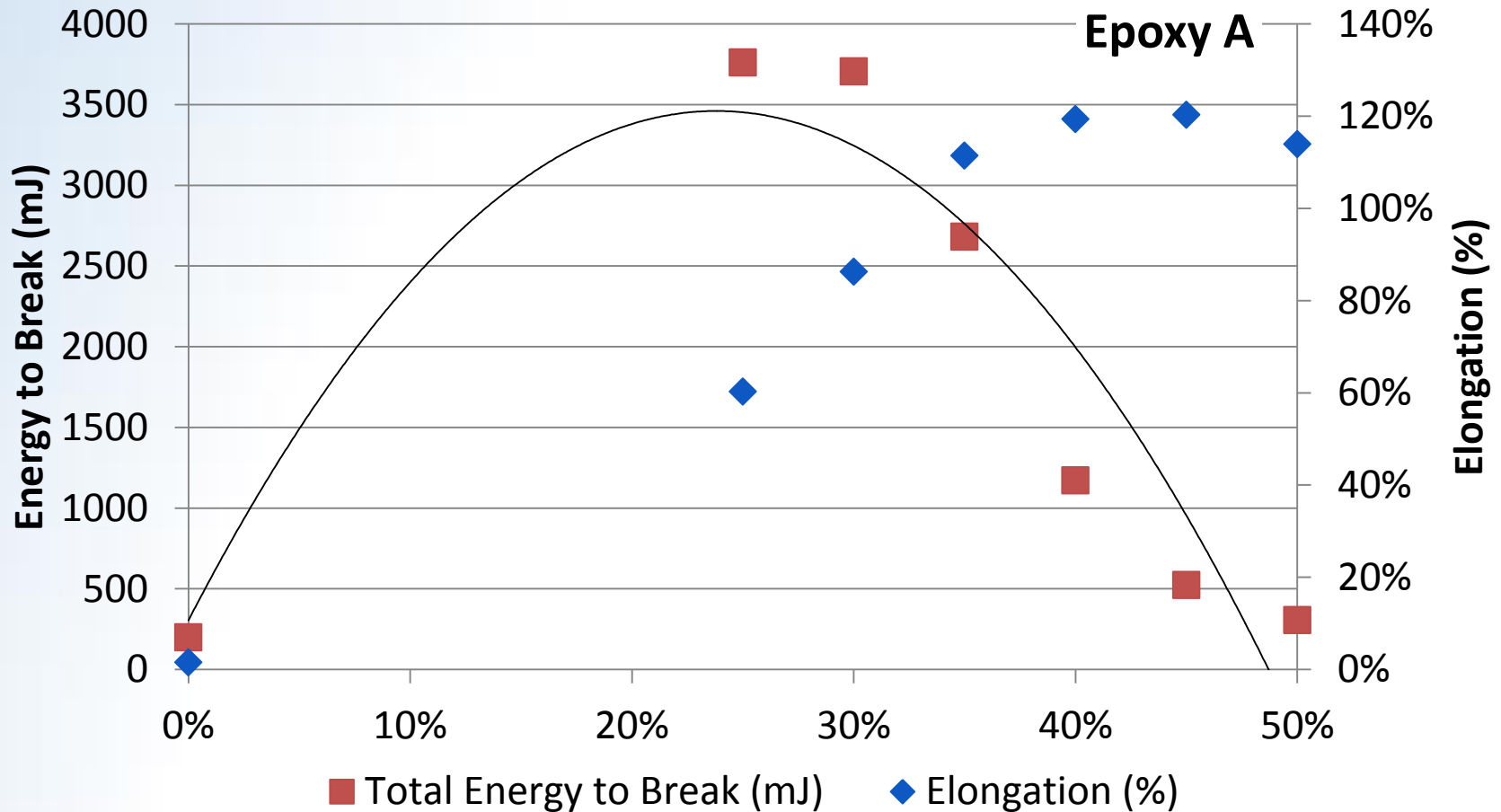
# Modulii



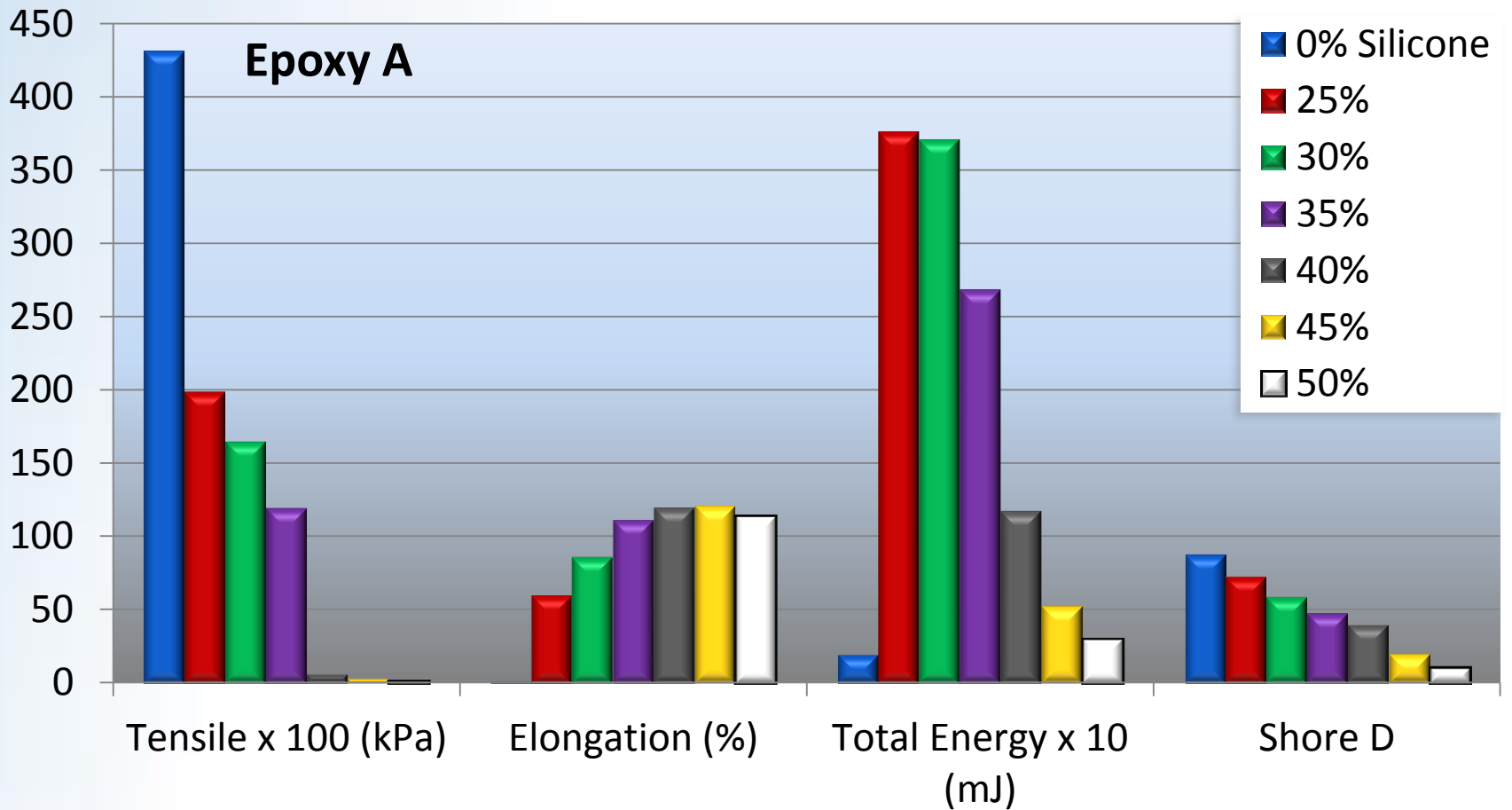
# Hardness and Strength



# Total Energy to Break/ Elongation



# Properties Epoxy A

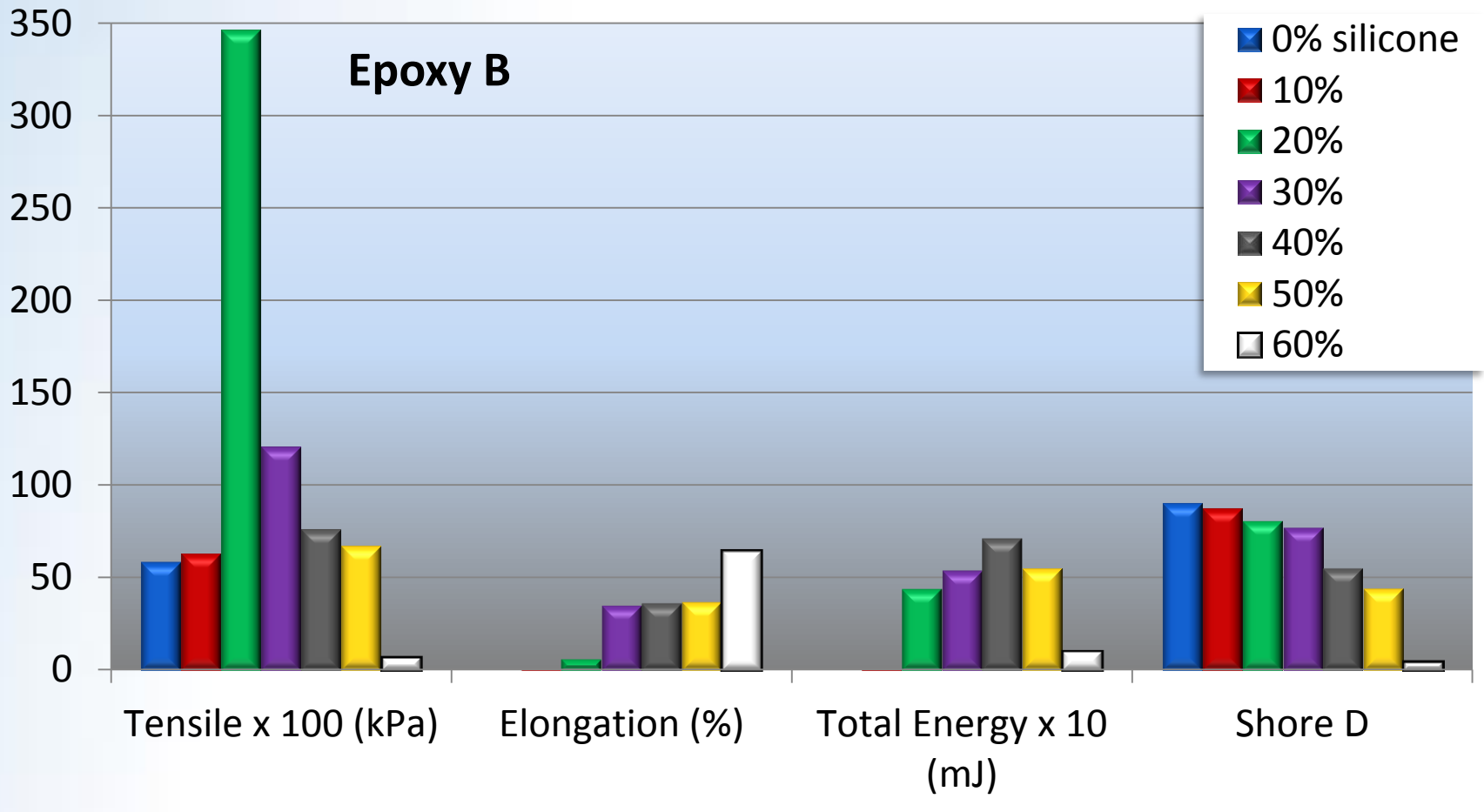


# Epoxy Silicones with Epoxy B

- ▶ UVACure 1500, a commercial cycloaliphatic epoxy system is reacted with epoxy silicone B
- ▶ MHPA and Imicure AM-1 used 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

# Amine Hardened Epoxy/ Silicones

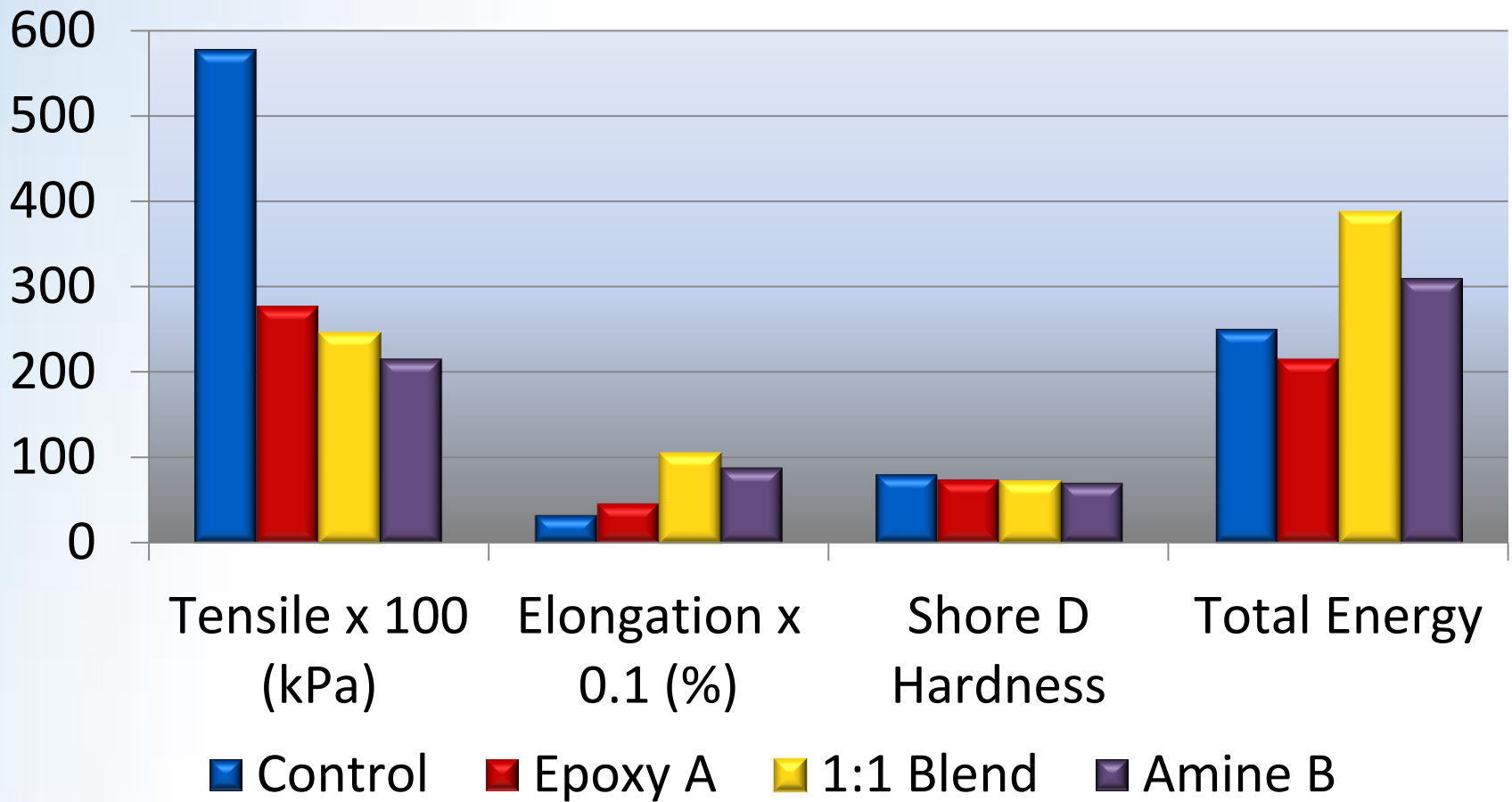
- ▶ Dow DER 331 Epichlorohydrin/ bisphenol A commercial system is reacted with reactive silicones
- ▶ MHPA and Imicure AM-1 used to affect cure.
- ▶ Cured at ambient for 24 hours.
- ▶ Ancamine 1618 is found to be best for hardening over Ancamine 1483 or TEPA.
- ▶ Properties are followed with Brookfield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

# Silicone Hardeners

Replace 20% of Ancamine 1618 (hardener):

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

# Results



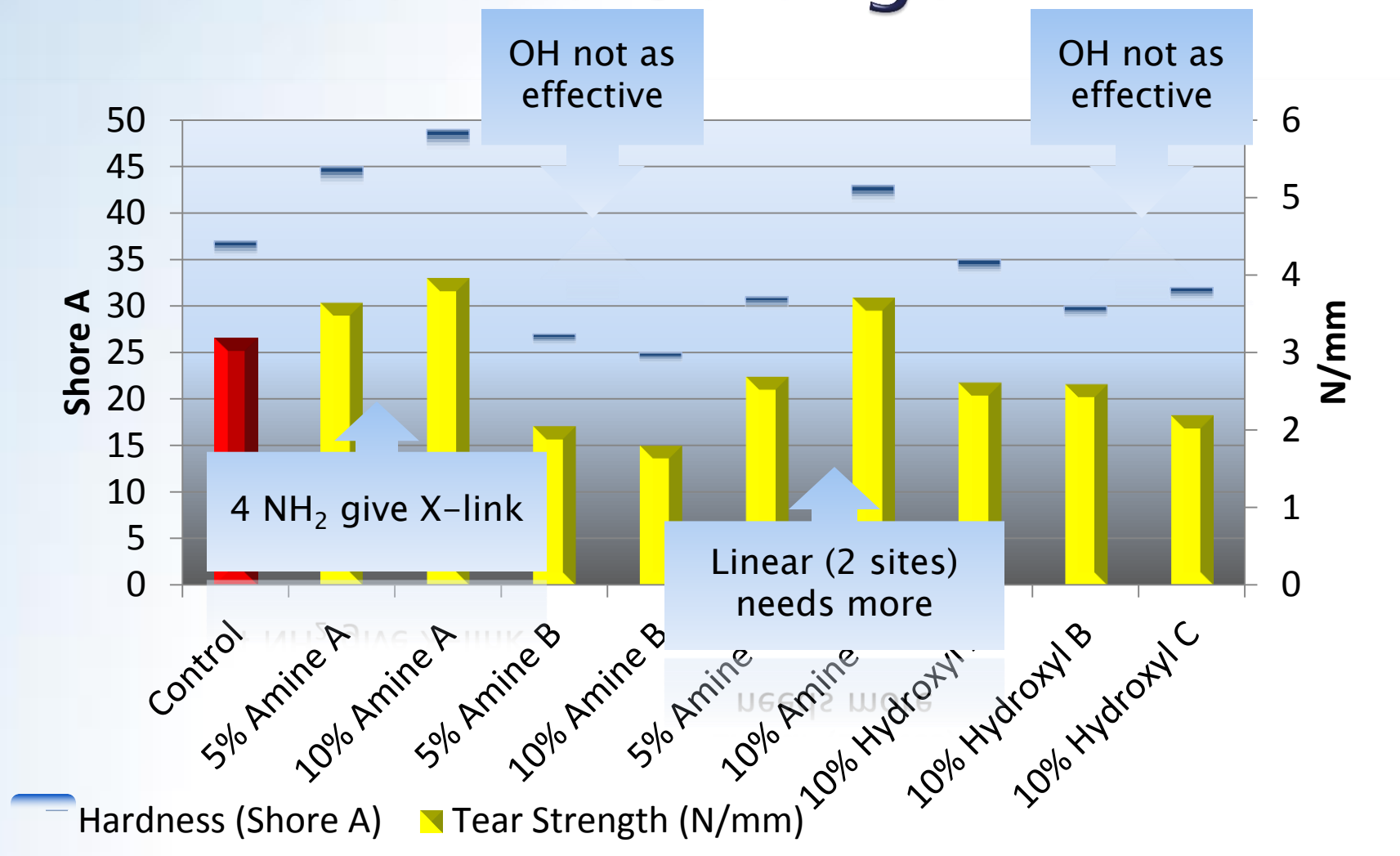
# Rubber Filled Epoxy with Silicone Hardener

- ▶ Proprietary epoxy with 5–10% reactive silicones and rubber crumbs
- ▶ Mold and cure at ambient for 7 days
- ▶  $-15^{\circ}\text{C}$  and  $-30^{\circ}\text{C}$  impact resistance
- ▶ Severity of fracture rated 1–10 (best)

# Results

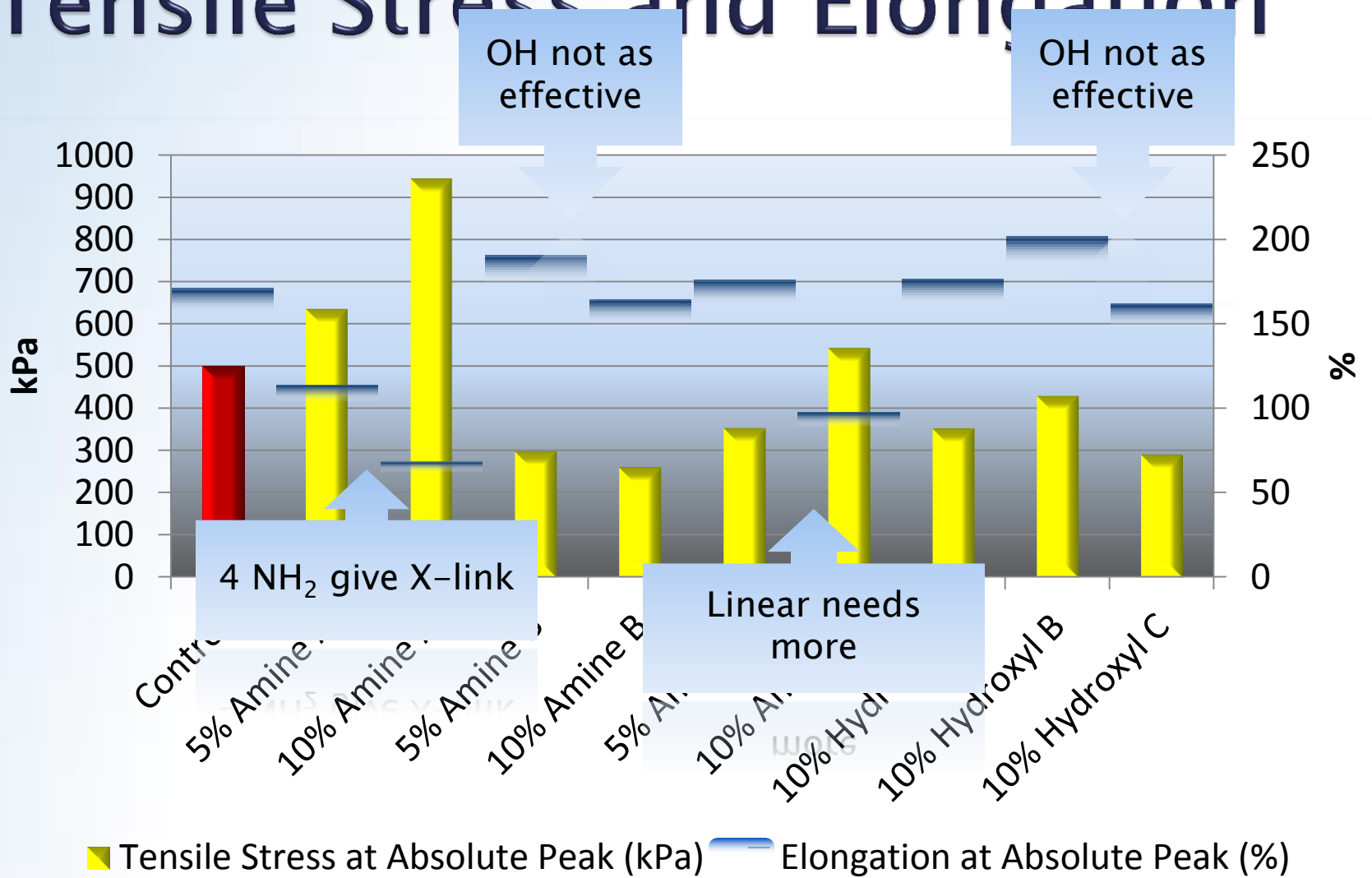
Additive	%	Shore A	Tear (N/mm)	Tensile (kPa)	Elongation (%)	- 15° C	- 30° C
Control	0	37	3.2	500	171	5	6*
Amine A	5%	45	3.6	636	114	8	7*
Amine A	10%	49	4.0	943	68	9.5	9*
Amine B	5%	27	2.1	299	191	5	5.5
Amine B	10%	25	1.8	261	164	4.5	2.5
Amine C	5%	31	2.7	354	176	4.5	3
Amine C	10%	43	3.7	543	98	9.5	8*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

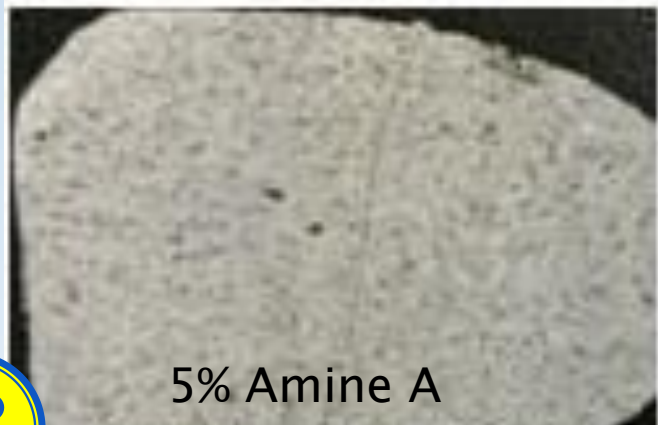




# Tensile Stress and Elongation



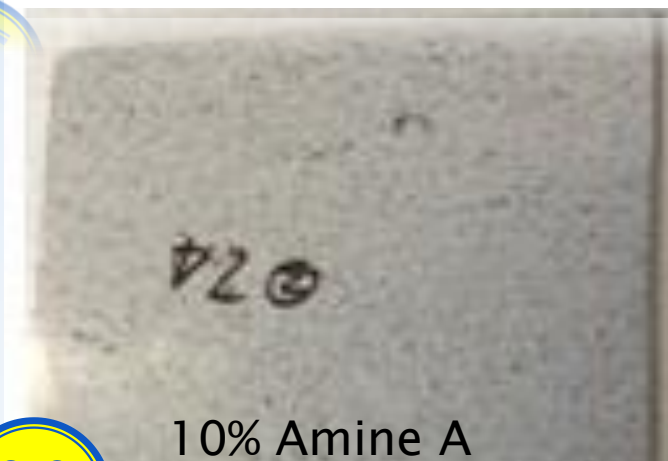
-15°C  
Fracture



5% Amine A



Control



10% Amine A

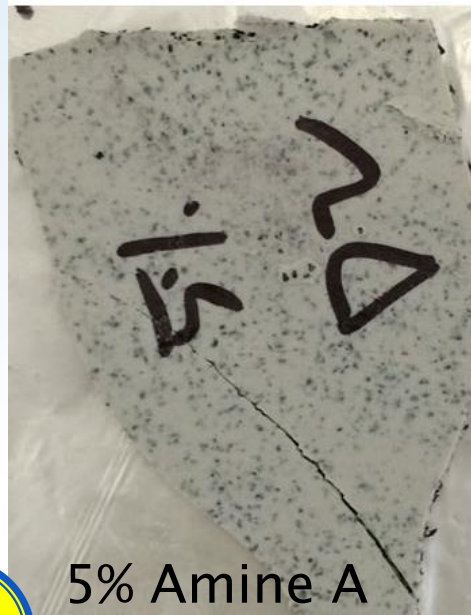


10% Hydroxyl A

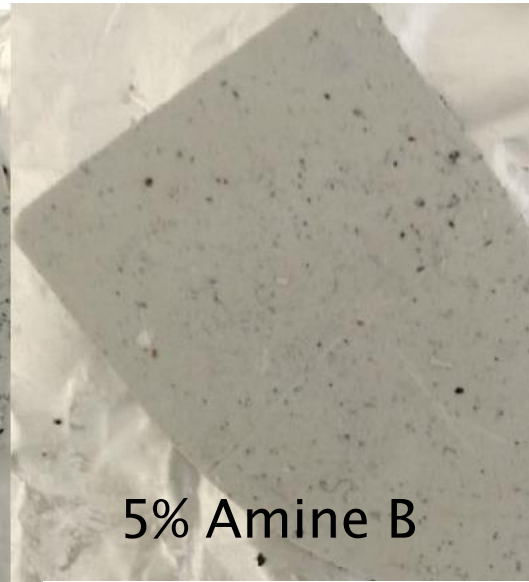




Control

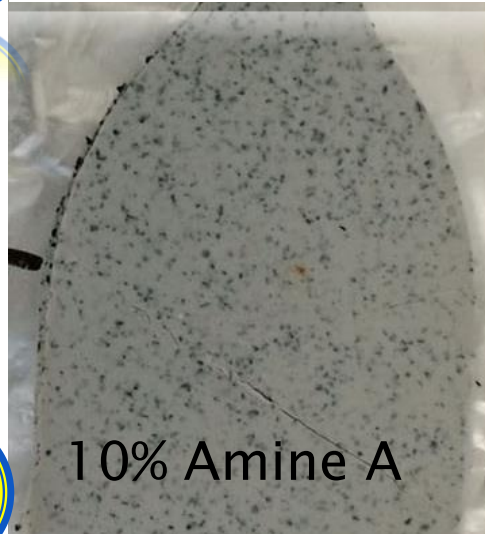


5% Amine A

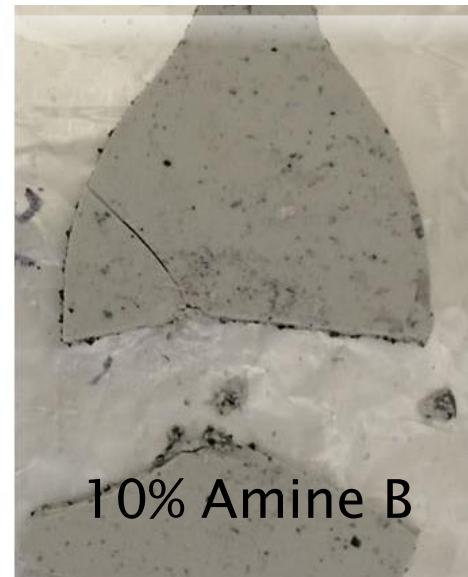


5% Amine B

-30°C  
Fracture



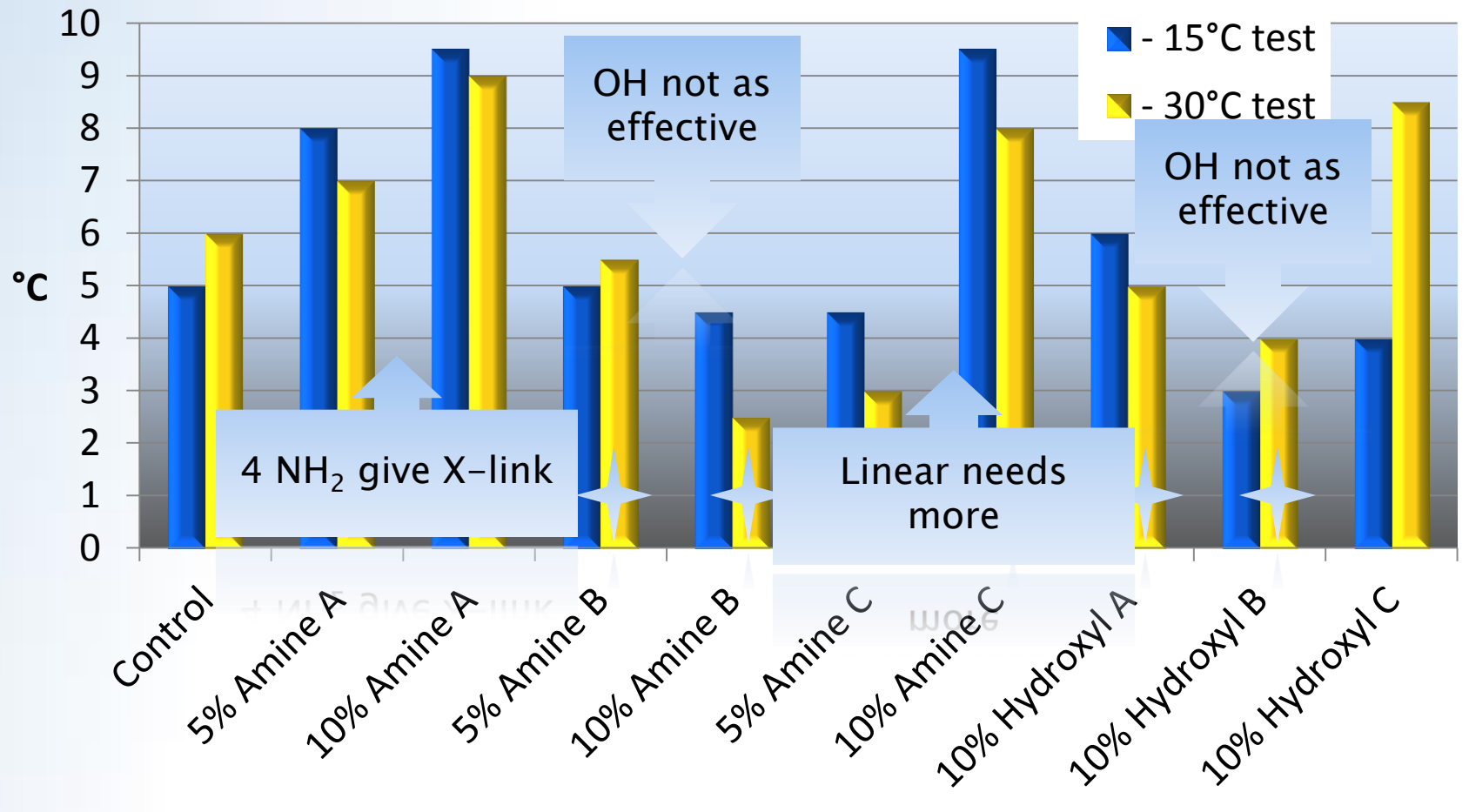
10% Amine A



10% Amine B



# Low Temperature Impact



★ Fractured on first of two impacts

# 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.