Static-Mixing Laboratory Techniques For Aliphatic Polyurea Coatings

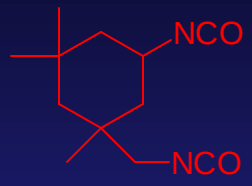
David W. House, Dejan Ilijevski, Ray Scott, and John Leon

Presented at the Polyurea Development Association

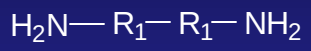
December 1, 2000

A Typical Formulation for a Sprayed, Aliphatic Polyurea Coating

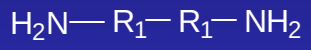
A-Side: Isophorone diisocyanate



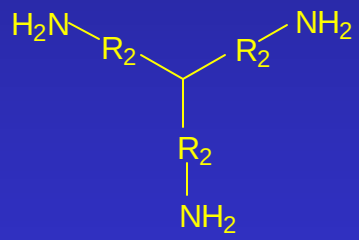
JEFFAMINE D-2000



B-Side: JEFFAMINE D-2000



JEFFAMINE T-5000



Curative(s)

Low molecular weight polyamine(s)

Benefits of Polyurea Coatings

* Improved physical properties
* Very fast cure time - fast turnaround time
* Fast physical property development (24h)
* Virtually unaffected by moisture
* Essentially unaffected by ambient temperature
* Do not require catalyst
* Better thermal stability

Challenges

* Very short pot life
* High formulation viscosity
  - Effective mixing
  - Application and adhesion to substrate
  - Surface uniformity

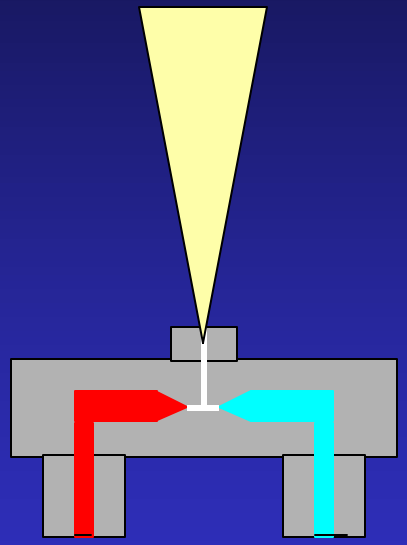
Solution

* Specialized equipment
* Formulation development

High-Pressure, Impingement Mixing

* Handles pot lives of 2+ seconds
* Heated components (160-170 ^{o}F)
* High pressures (2000-3000 psi)
* Uses spray gun to continuously deliver the atomized formulation
* Component volumes in gallons
* Excellent in field use (large-scale)

High-Pressure Impingement Mixing



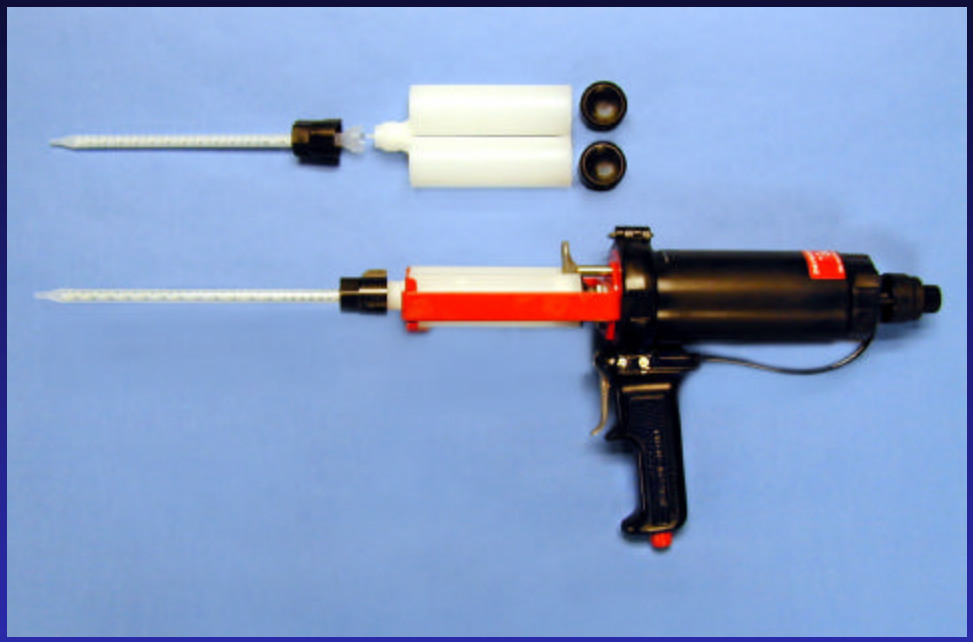
Challenge

* Lab-scale formulation development
  - Equipment size
  - Component volumes
  - Change-over time

Solution Requirements

* Screen large number of formulations in a short time
* Mimic field equipment (impingement mixing)
* Handle the very fast reaction kinetics

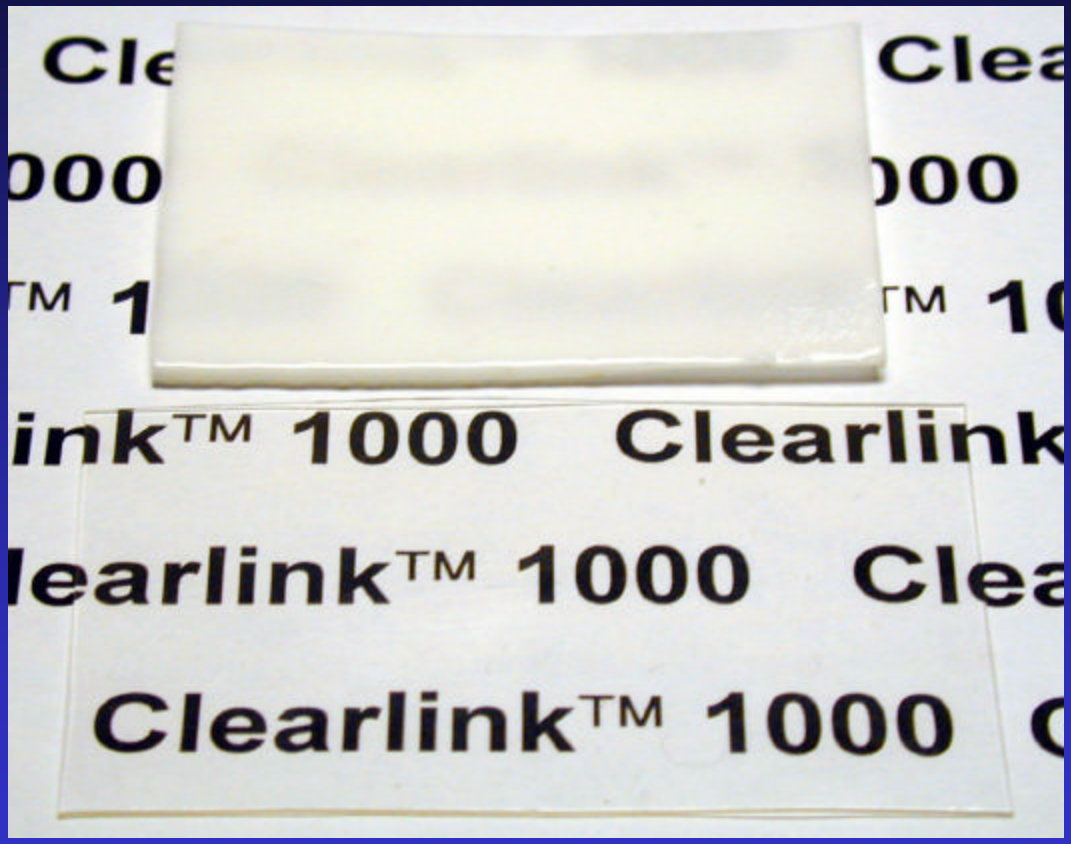
Static Mix Gun Assembly



Static (Motionless) Mixing

* Uses extrusion to static mix the components and a draw down knife to spread the coating
* Low pressures (about 80 psi)
* Room temperature and higher
* Component volumes in grams
* Fast, simple clean-up (disposable cartridges)
* Quick screening of many different formulations
* Inexpensive (equipment and chemicals)
* Small, uses minimal lab space
* Uses same basic formulation parameters as those used in impingement mixing
* Technique is easy to learn
* Handles pot lives of 10+ seconds

Coatings Made Using Impingement Mixing versus Static Mixing



Clearlink 1000

Cycloaliphatic Secondary Diamine

• Clear, colorless liquid	
• Equivalent Weight	161
• Specific Gravity	0.90 at 68°F (20°C)
• Viscosity	36 cSt at 100°F (38°C)
• Pour Point	-44°F (-42°C)
• Flash Point (PMCC)	325°F (163°C) min
• Vapor Pressure	< 1 mm Hg at 68°F (20°C)
• Ames Test	Nonmutagenic
• LD_{50} (acute oral, rat)	482 mg/kg

Comparison of Static Mixing to Impingement Mixing

Formulation	Impt Mixing	Static Mixing	Static Mixing
A-Side			
Desmodur I (%)	45	44.6	39.2
JEFFAMINE D-2000 (%)	55	55.4	60.8
% NCO	14.8	14.5	12.2
Index	105	105	105
B-Side			
JEFFAMINE D-2000 (%)	32.2	35.0	49.3
JEFFAMINE T-5000 (%)	8.6	15.2	12.7
Clearlink 1000 (%)	46.8	49.8	38.0
TiO_2 (%)	12.4	0.0	0.0

Comparison of Static Mixing to Impingement Mixing

Processing	Impt Mixing	Static Mixing	Static Mixing
Volume Ratio (A/B)	1.00	1.00	1.00
Weight Ratio (A/B)	0.96	1.08	1.08
Spray/Cartridge Temp. (^{o}F)	140	73	73
Coating Thickness (mil)	41	31	34
Pot Life (sec)	9	~ 15	< 20
Tack-Free Time (min)	---	3.5	15

Comparison of Static Mixing to Impingement Mixing

Physical Properties	Impt Mixing	Static Mixing	Static Mixing
Hardness, inst. (Shore D)	43	60	48
Tensile Strength (psi)	2820	4060	4170
100% Modulus (psi)	1350	1560	1040
Elongation (%)	500	530	600
Tear Resistance (pli)	500	630	490
Tear Prop. Resist. (pli)	525	550	400
Resilience (%)	40	41	40

Comparison of Static Mixing to Impingement Mixing

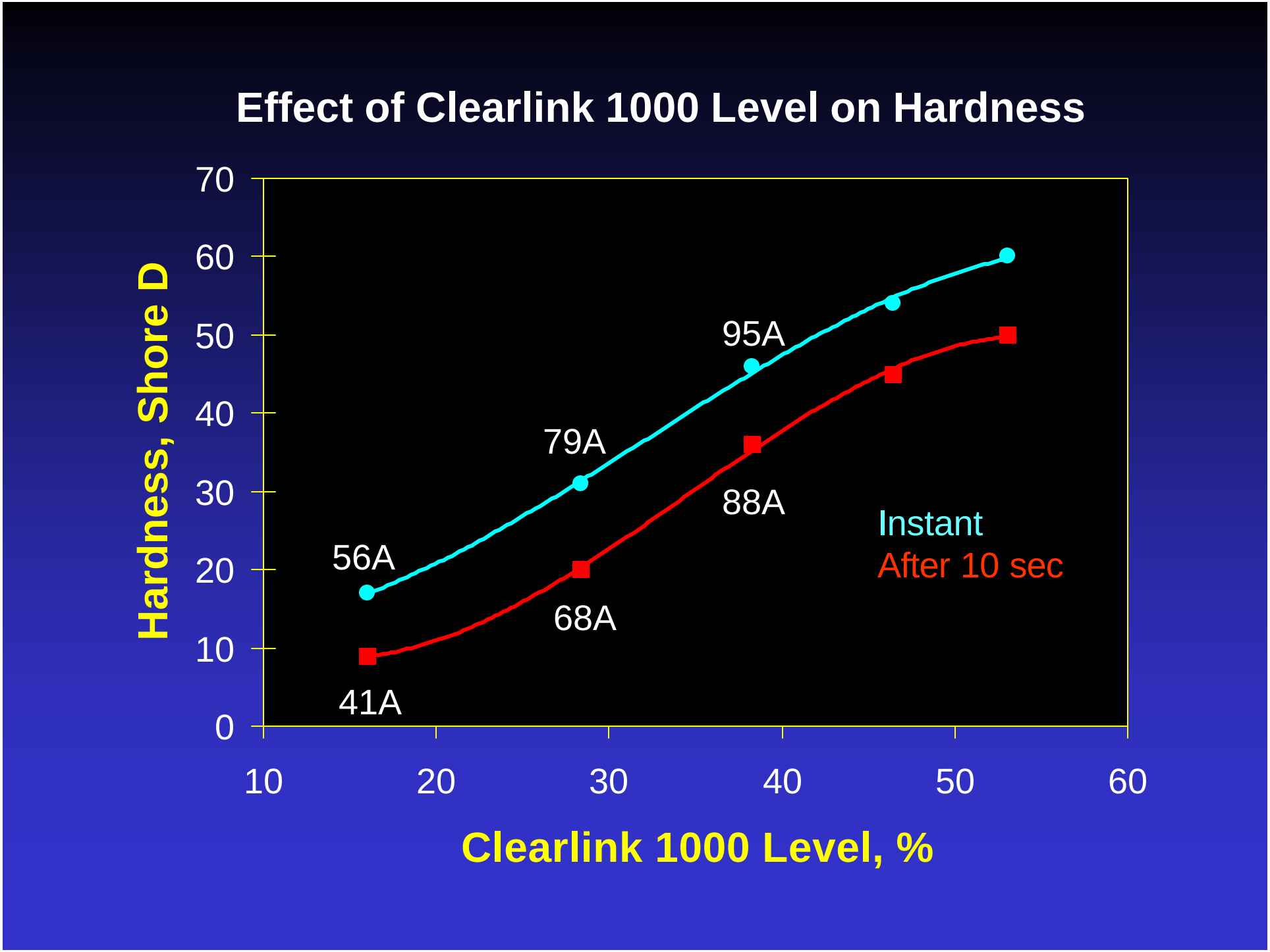
Formulation	Impt Mixing	Static Mixing
A-Side		
Desmodur I (%)	45	39.6
JEFFAMINE D-2000 (%)	55	60.4
% NCO	14.8	12.4
Index	105	105
B-Side		
JEFFAMINE D-2000 (%)	40.7	50.7
JEFFAMINE T-5000 (%)	9.9	12.8
VESTAMIN IPD	7.7	2.0
Clearlink 1000 (%)	30.8	34.5

Comparison of Static Mixing to Impingement Mixing

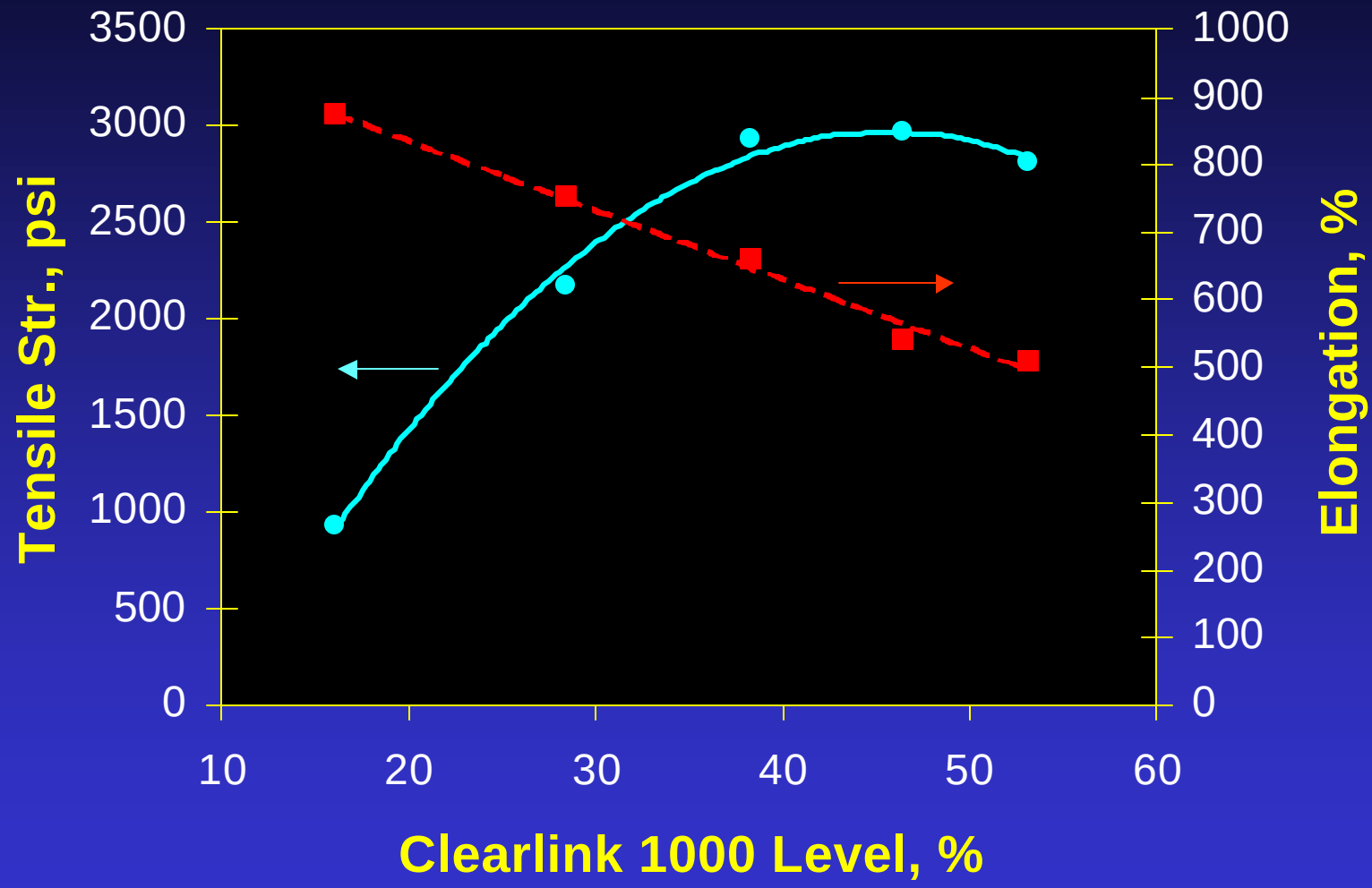
Physical Properties	Impt Mixing	Static Mixing
Hardness, inst. (Shore D)	42	48
Tensile Strength (psi)	2510	4340
100% Modulus (psi)	1200	1075
Elongation (%)	470	600
Tear Resistance (pli)	415	500
Tear Prop. Resist. (pli)	345	390
Resilience (%)	39	45

Effect of Clearlink 1000 Level on Polyurea Coatings

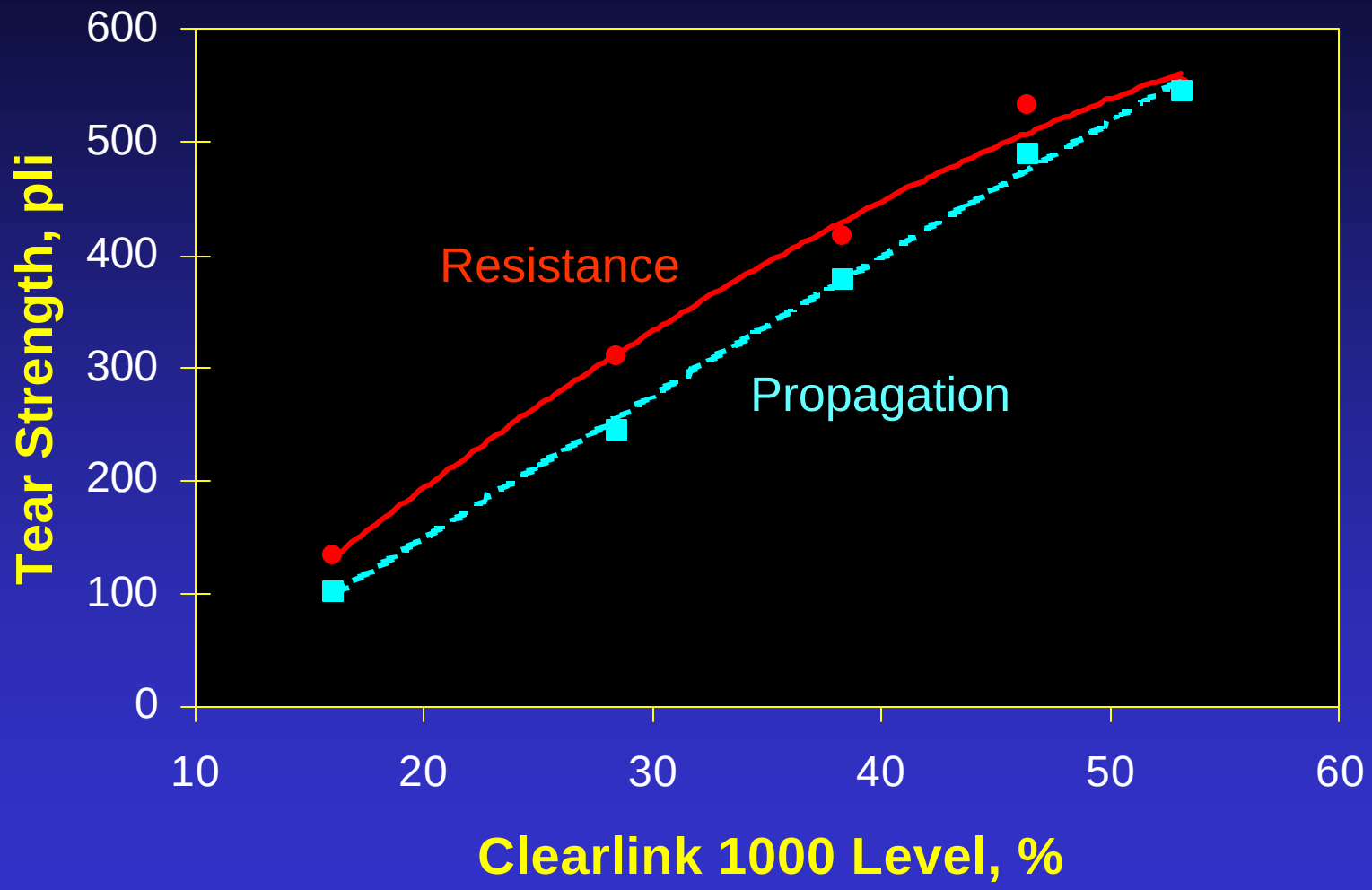
A-Side					
Desmodur I (%)	26.7	33.1	38.1	42.1	45.0
JEFFAMINE D-2000 (%)	73.3	66.9	61.9	57.9	55.0
% NCO	7.0	9.7	11.8	13.4	14.8
Index	105	105	105	105	105
B-Side					
JEFFAMINE D-2000 (%)	36.1	29.1	23.4	18.8	15.0
JEFFAMINE T-5000 (%)	47.9	42.6	38.3	34.8	31.9
Clearlink 1000 (%)	16.0	28.4	38.3	46.4	53.1
Volume Ratio (A/B)	1.00	1.00	1.00	1.00	1.00
Weight Ratio (A/B)	1.03	1.05	1.06	1.08	1.09
Coating Thickness (mil)	34	33	33	36	28
Pot Life (sec)	< 30	< 30	< 30	< 30	< 30
Tack-free Time (min)	2.5	4.0	6.0	9.0	> 10
Hardness, instant (Shore D)	17 (56A)	31 (79A)	46	54	60
After 10 sec (Shore D)	9 (41A)	20 (68A)	36	45	50


Effect of Clearlink 1000 Level on Hardness
Hardness, Shore D
70
60
50
40
30
20
10
0
10
20
30
40
50
60
Clearlink 1000 Level, %
56A
79A
95A
41A
68A
88A
Instant
After 10 sec

Effect of Clearlink 1000 Level on Tensile Strength and Elongation



Effect of Clearlink 1000 Level on Tear Strength

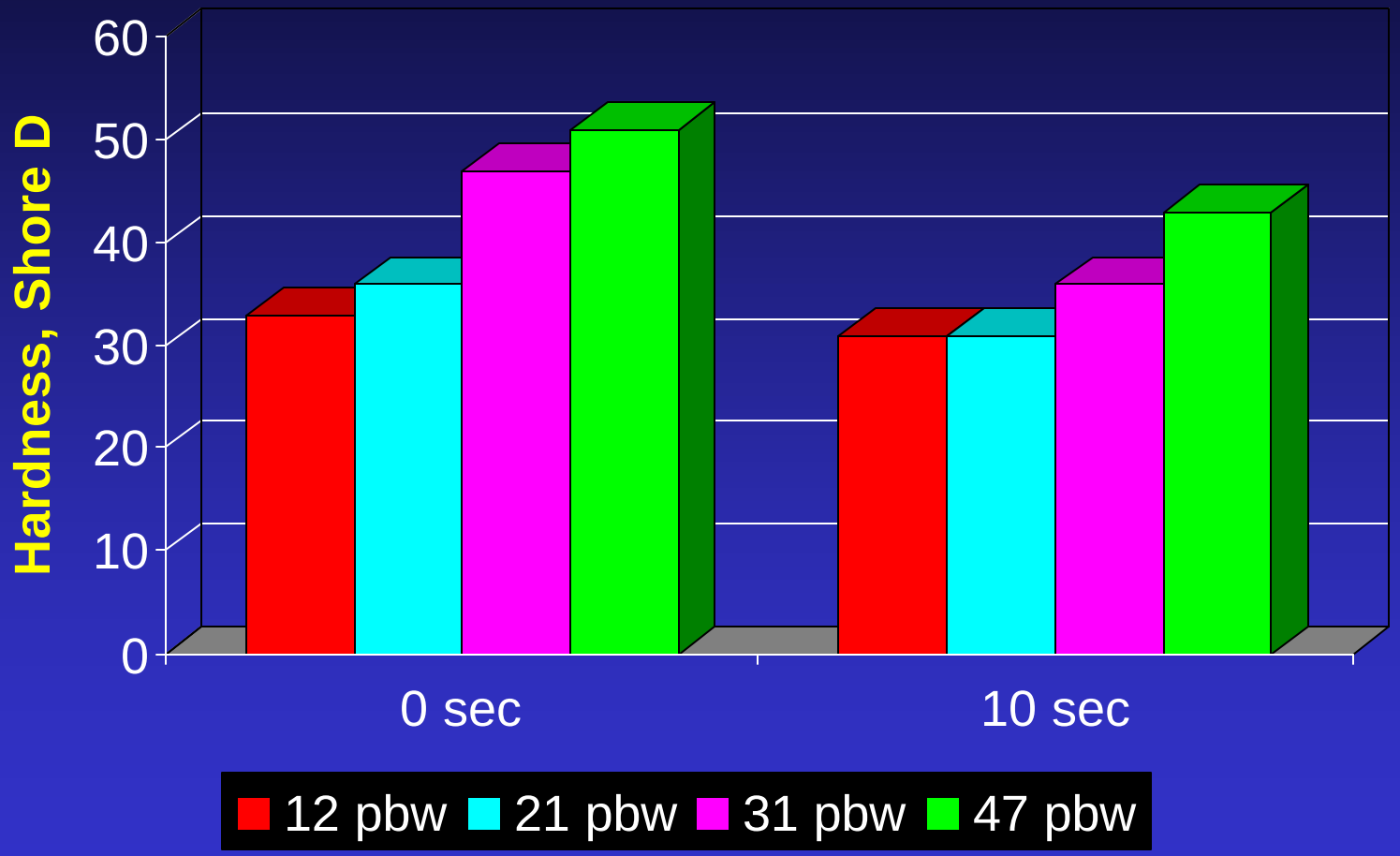


Formulations for the Chain Extender Level Study

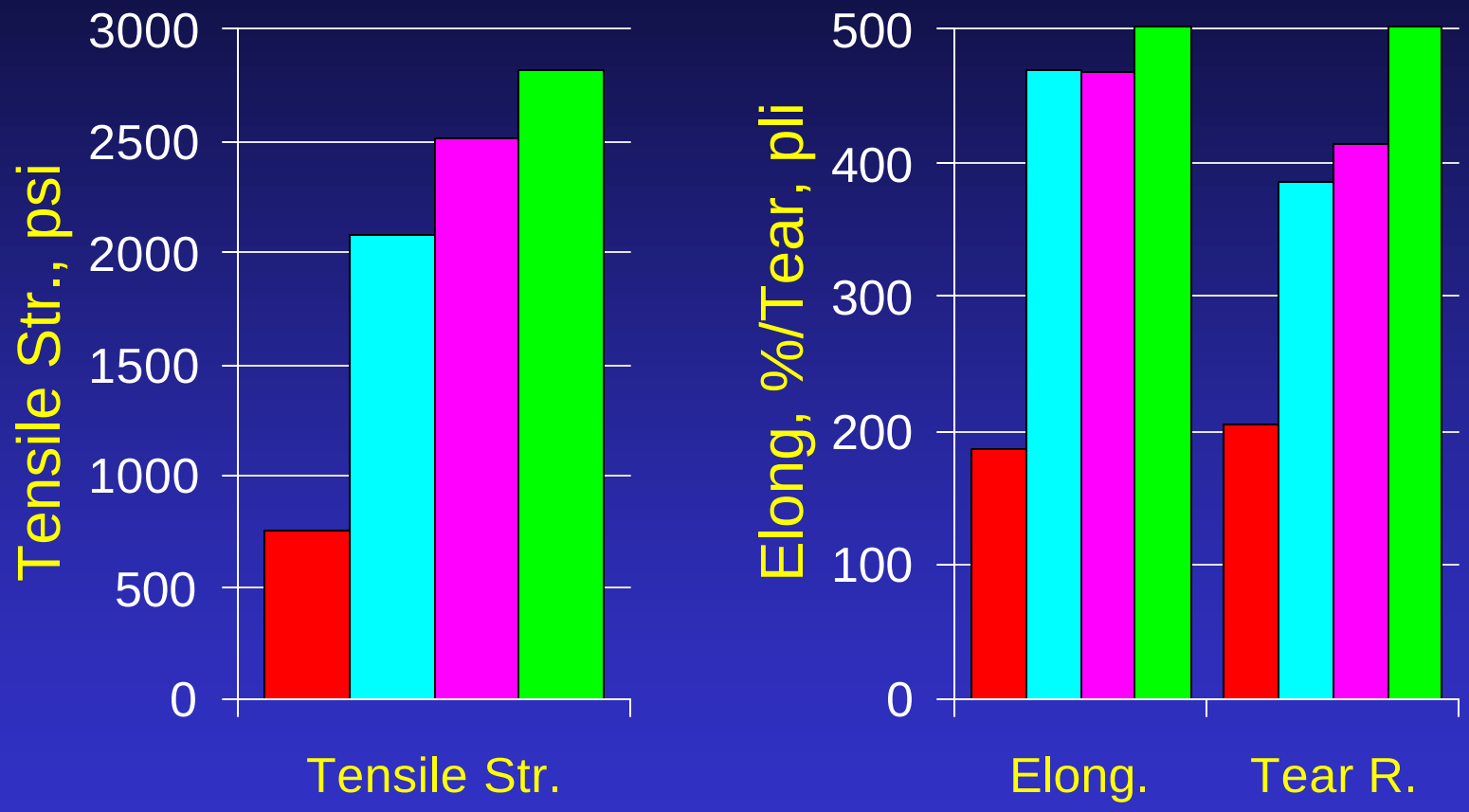
Isocyanate Component	A	B	C	D	E
VESTANAT® IPDI	45	45	45	45	45
JEFFAMINE® D-2000	55	55	55	55	55
%NCO	14.8	14.8	14.8	14.8	14.8
Resin Blend					
JEFFAMINE® D-2000	48.17	48.62	45.61	40.66	32.18
JEFFAMINE® T-5000	14.96	12.15	10.01	9.89	8.60
VESTAMIN® IPDA	22.83	16.57	11.96	7.69	0.00
Clearlinkâ 1000	*0.0*	*11.60*	*21.02*	*30.77*	*46.78*
Ti02	10.64	11.05	11.12	10.00	12.38

Index = 105, Iso/Resin Ratio = 1.00:1.00

Effect of Clearlink 1000 Level on Physical Properties



Effect of Clearlink 1000 Level on Physical Properties



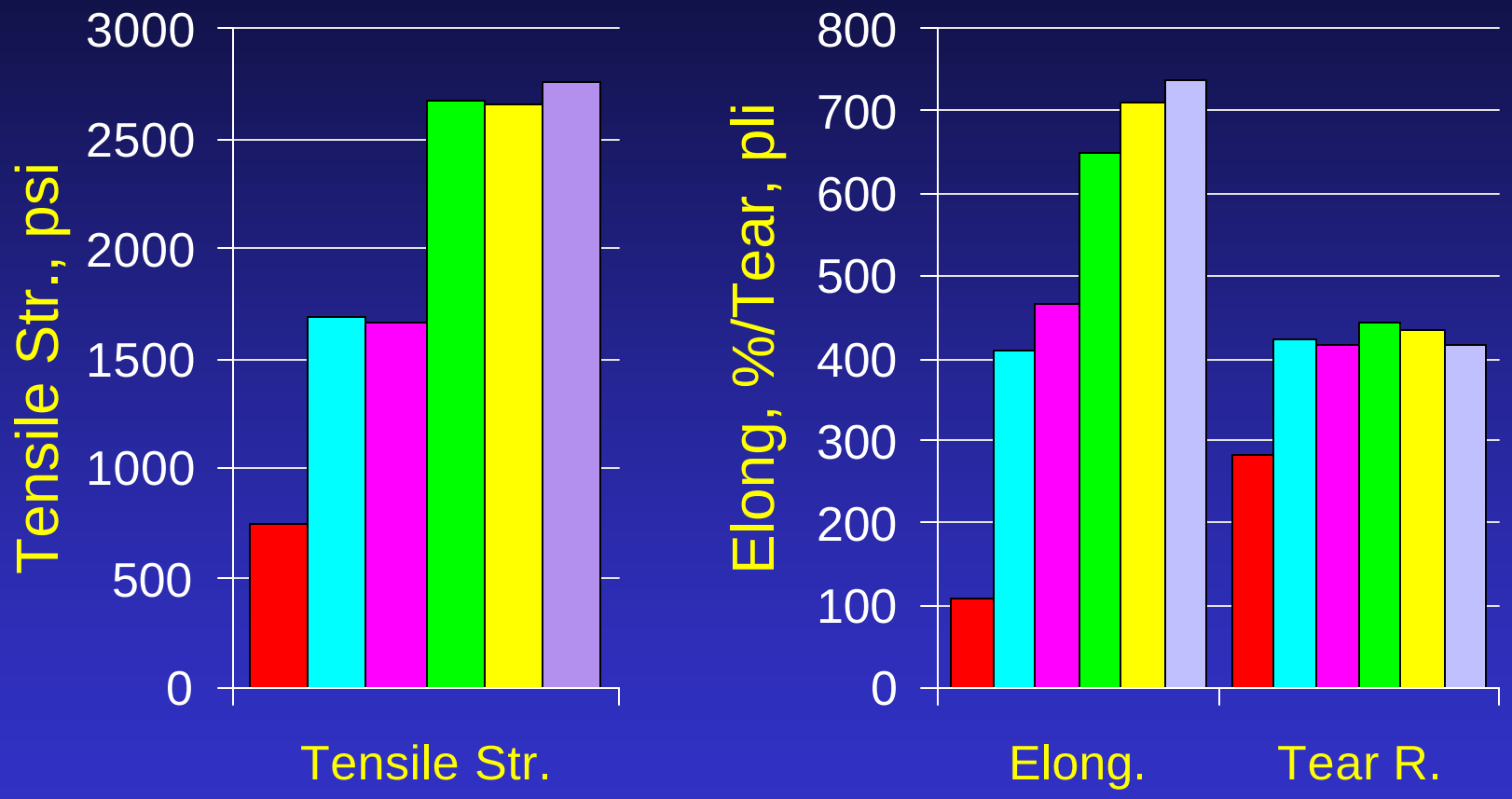
Pbw Clearlink 1000: 12, 21, 31, 47.

Effect of Isocyanate Index on Physical Properties

Isocyanate Index	0.95	1.00	1.05	1.10	1.15	1.20
Isocyanate Component						
VESTANAT IPDI	45	45	45	45	45	45
JEFFAMINE® D-2000	55	55	55	55	55	55
%NCO	14.8	14.8	14.8	14.8	14.8	14.8
Resin Blend						
JEFFAMINE D-2000	43.21	44.70	45.83	46.67	47.25	48.65
JEFFAMINE T-5000	15.43	15.59	16.67	17.78	18.68	19.46
VESTAMIN IPDA	15.43	13.51	12.50	11.11	9.89	9.73
Clearlink 1000	25.93	26.20	25.00	24.44	24.18	22.16

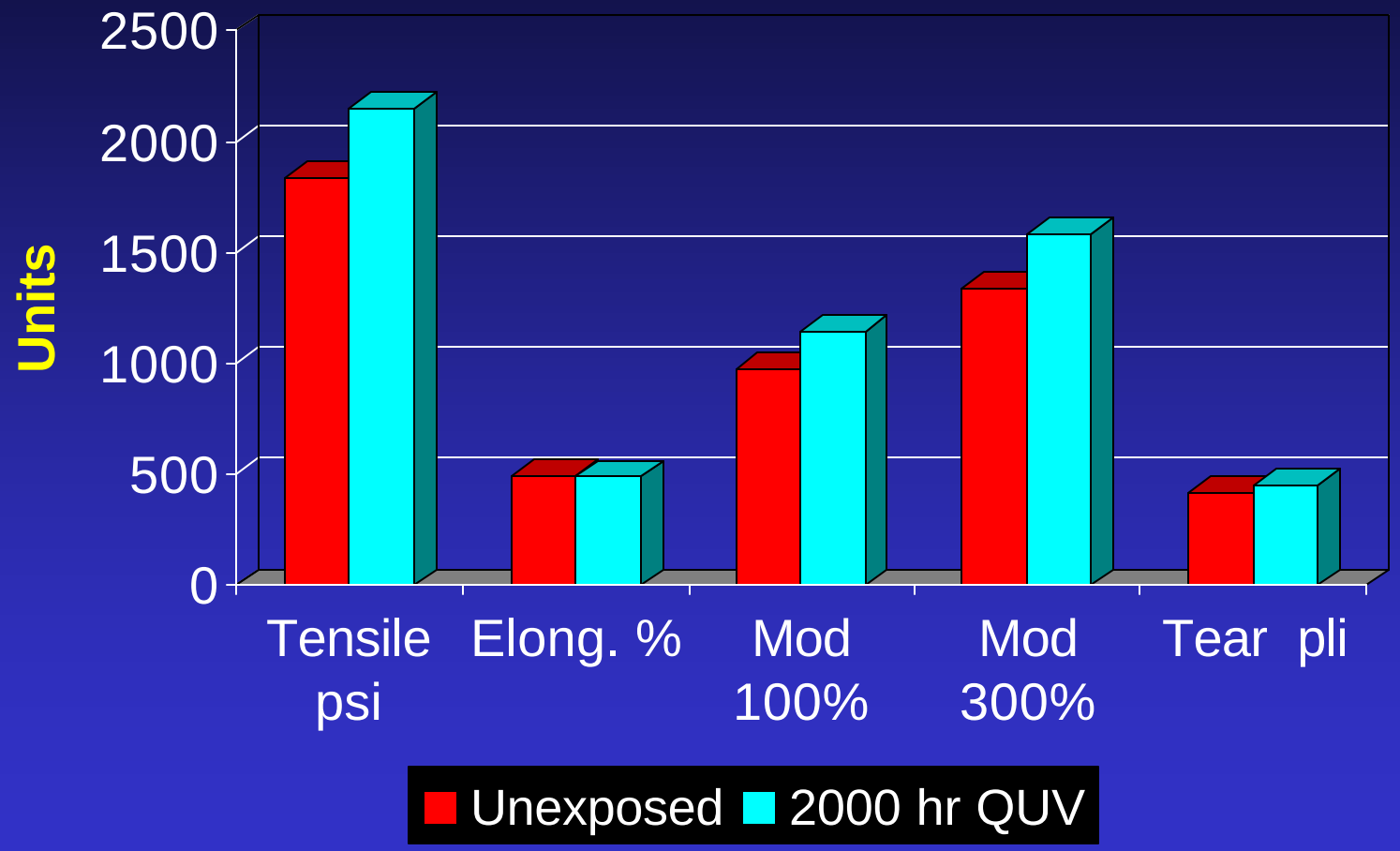
Iso/Resin Ratio = 1.00:1.00

Effect of Isocyanate Index on Physical Properties: ~25 pbw CL1000, 41 Shore D

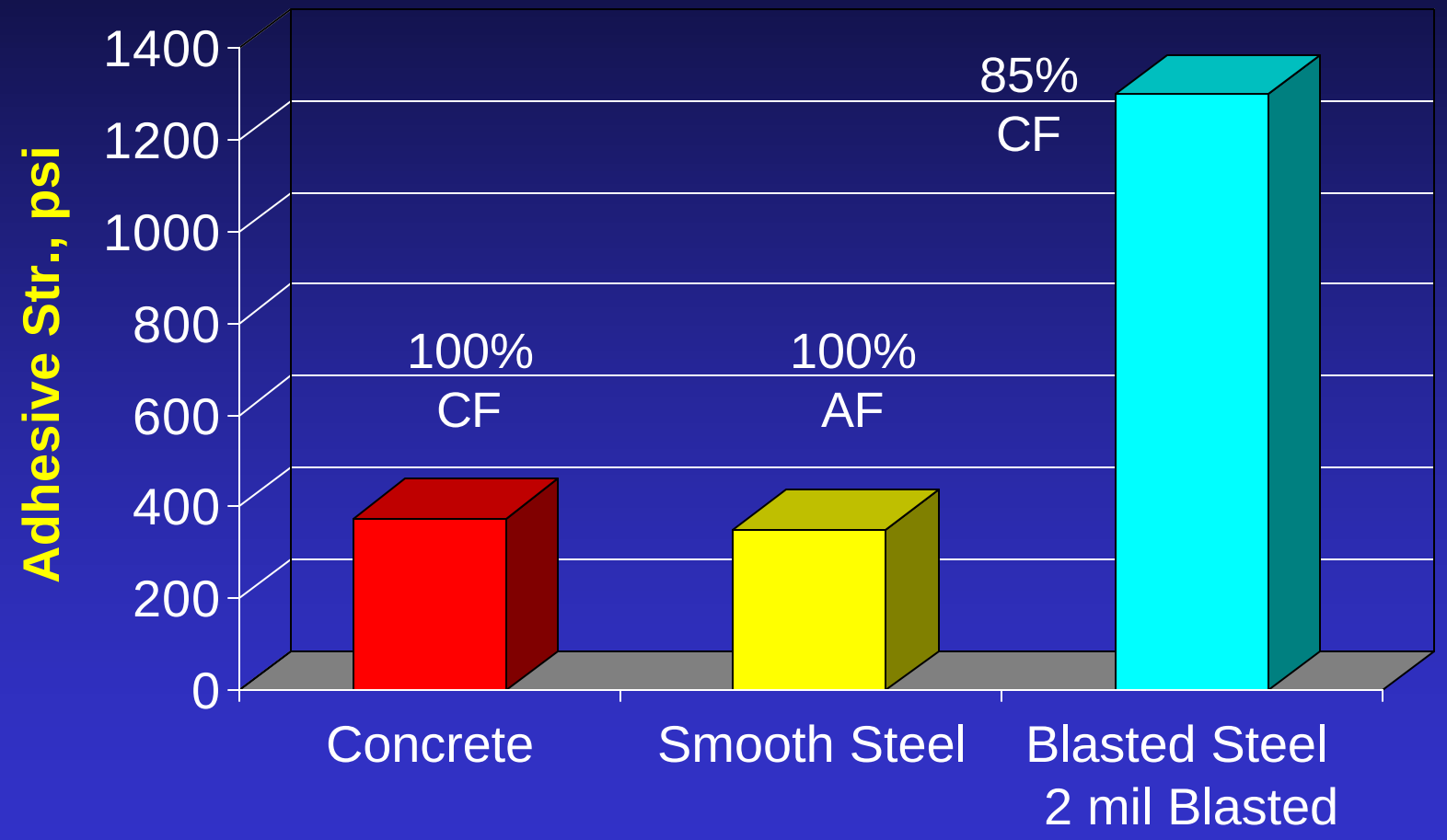


Index: 95, 100, 105, 110, 115, 120

QUV Weatherometer Study
2000 Hours Exposure to a UV B-313 Bulb



Elcometer Adhesion Strength of a Clearlink 1000-Based Polyurea Coating



Conclusions

* A new laboratory technique has been developed for screening polyurea and polyurethane impingement-mixed formulations

* The technique mimics the physical properties of coatings made using impingement mixing and is useful for establishing trends

* The static mixing method is fast, easy to learn, inexpensive, requires no specialized equipment or lab preparation, and needs minimal space.

Summary

The use of Clearlink 1000 in polyurea coatings...

* Produces light-stable coatings
* Increases working time
* Improves adhesion to substrate

Significantly increases:

* Hardness
* Tensile strength
* Elongation
* Tear Strength

Acknowledgement

Aureliano Perez, Jr. and Kenneth M. Hillman
of Huntsman Corporation, USA

“It just sprays better”