

Effect of Physical and Chemical Aging on the Yield Stress of the filled Epoxy System 828DEAGMB

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Abstract

- Instron compression testing of the aging of 828DEA/GMB in relation to 828DEA
- Epoxy: Epon 828 (known also as DGEBA)
- Curative: Diethanolamine (DEA)
- Filler: 3M D32-4500 glass microballoons (GMB)
- Temperatures used: 55C, 60C, 65C, 70C, 75C
- Aged up to 2000 hours
- Compare to unfilled system
- Some 828DEA samples aged under stress

Sample Preparation



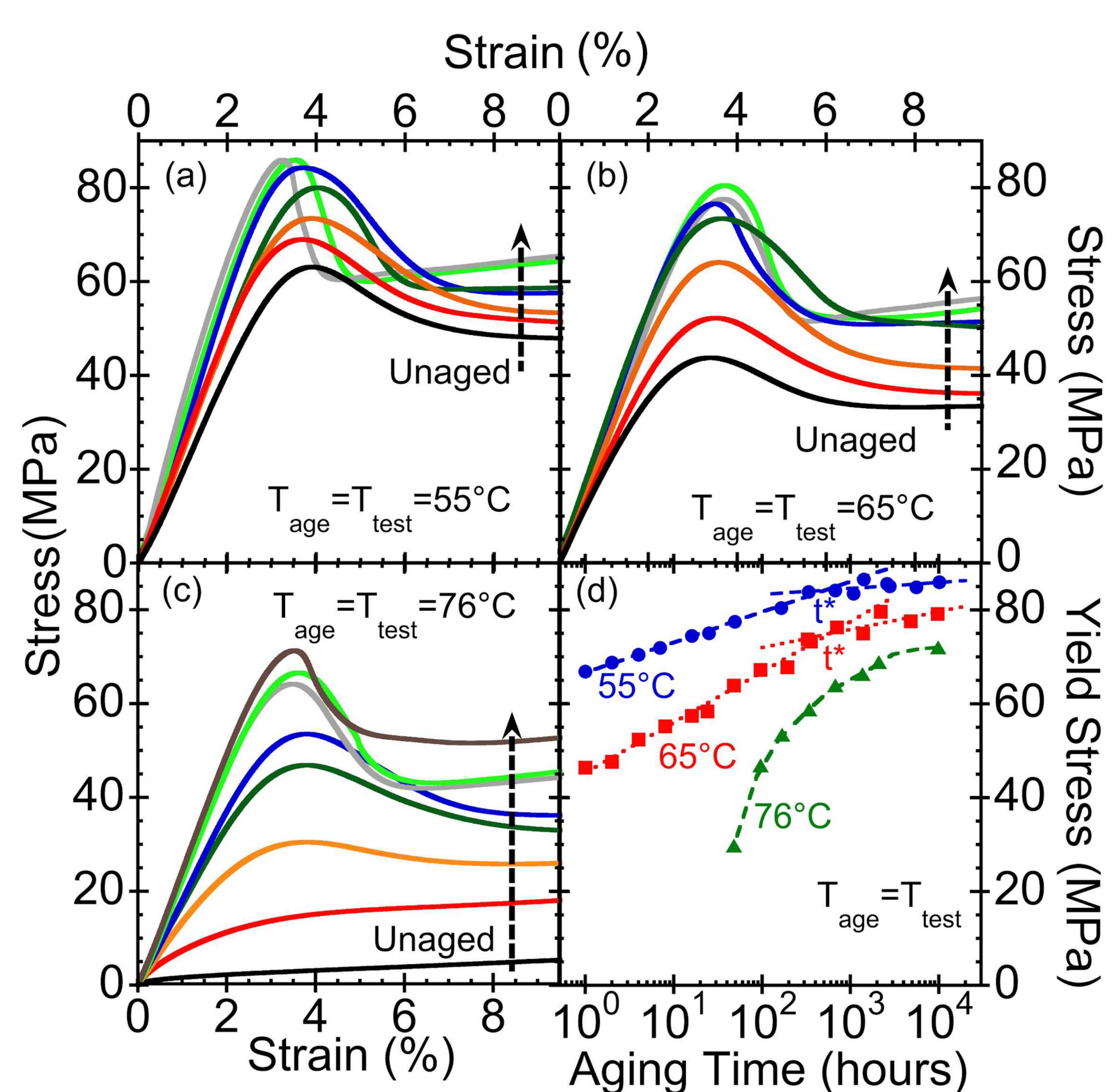
- Epon 828, D32-4500 glass microballoons (GMB), and diethanolamine (DEA) are mixed together
- Solution is degassed in vacuum oven at 70 C and 7 torr
- Placed into 10 mL syringes
- Cured at 70 C for 24 hours
- Cut into cylinders
- Sample size: 1 in. tall, 0.56 in. diameter
- Epoxy then annealed to remove thermo-mechanical history gained during processing
- Cooled at 1°C/min
- Samples designated for aging are then placed in ovens for predetermined amount of time
- Oven temperatures used: 55 C, 60 C, 65 C, 70 C, and 75 C

Testing Method

- Compression tests performed on an Instron 1125R at aging temperature
- Samples are equilibrated for 30 minutes prior to testing, then pre-loaded to ~20 lbs
- Compressed beyond its yield point
- Compression rates used: 0.1 ipm, 0.01 ipm, 0.001 ipm, and 0.0001 ipm
- Aging under stress performed while compressed at 40 MPa

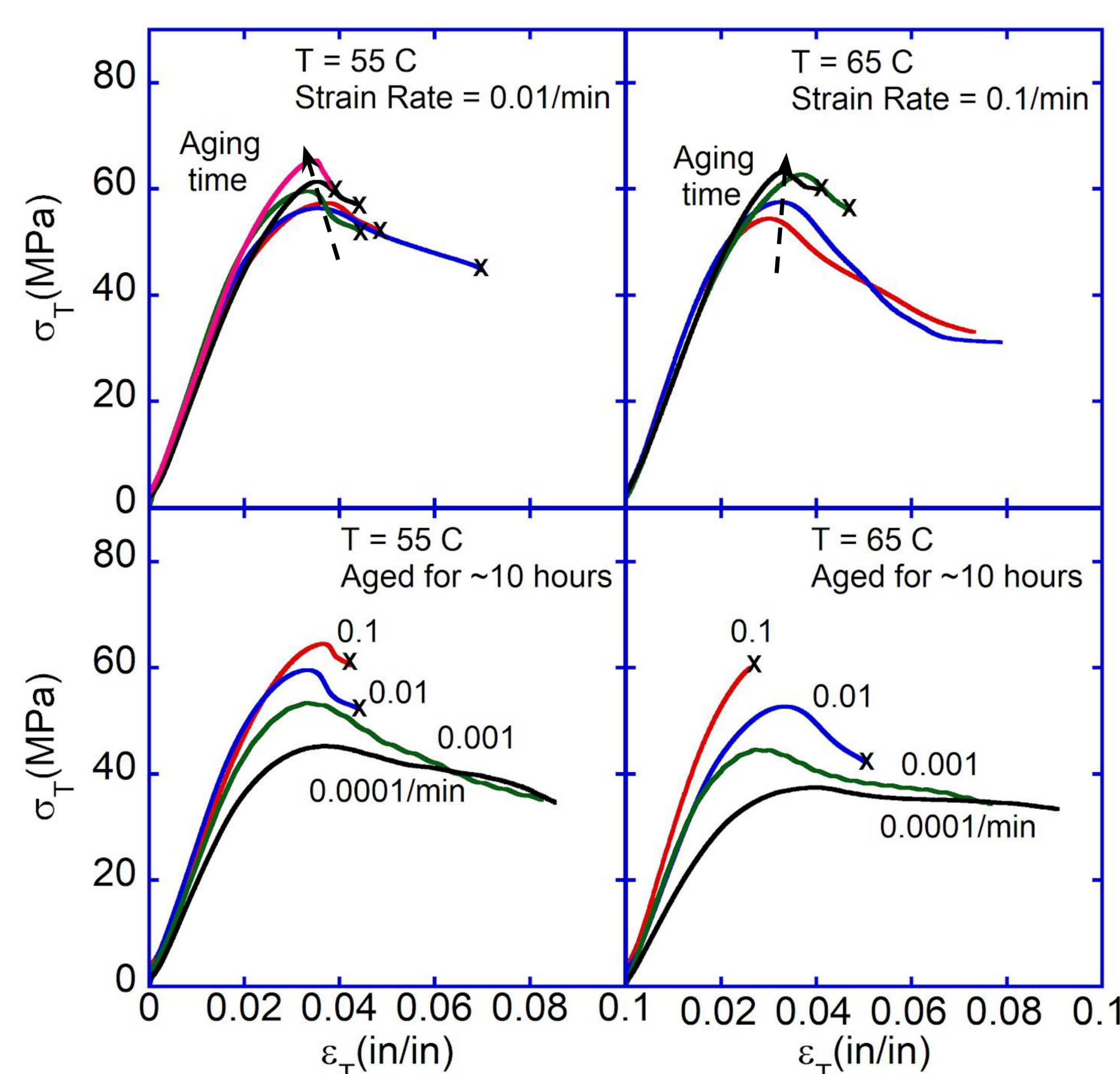


Unfilled σ_Y [1]



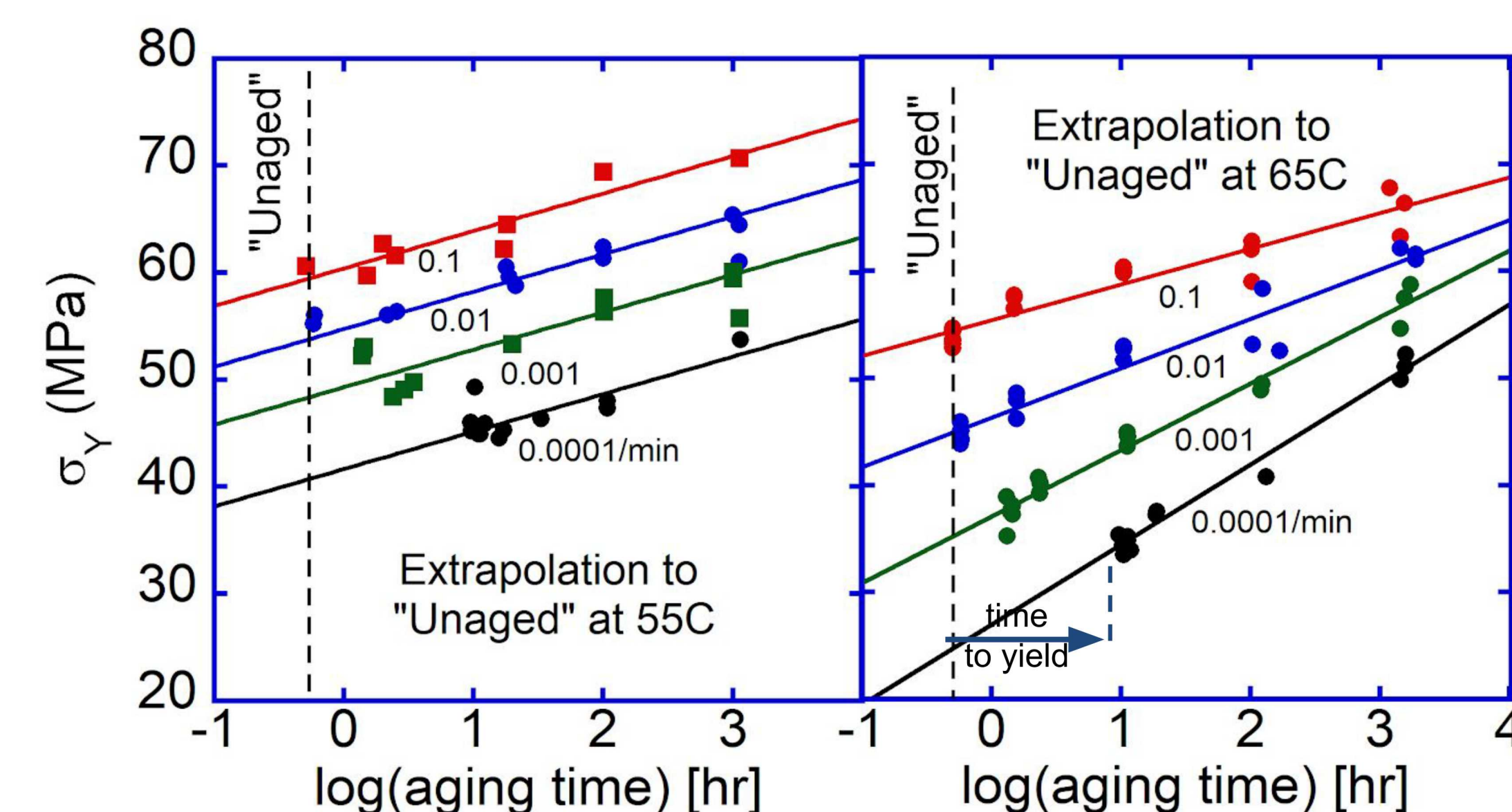
- From Arechederra et. al [1]
- Increase of yield stress linear with log(aging time)
- In keeping with a reduction of free volume
- Ductile yield in all cases
- Narrowing of yield peak with aging time
- Effect of both Chemical and Physical aging

Glass Microballoon Filled σ_Y



- Tests at 55 C and 65 C performed so far
- Mix of ductile and brittle failures

GMB Filled: The "Unaged" State



- Samples continue to age during testing
- "Unaged" state found through linear extrapolation
- Constant slopes at 55°C suggest constant V_a while 65°C shows an increasing V_a

σ_Y as function of t_{age} and strain rate

From Ree-Eyring Theory [2]:

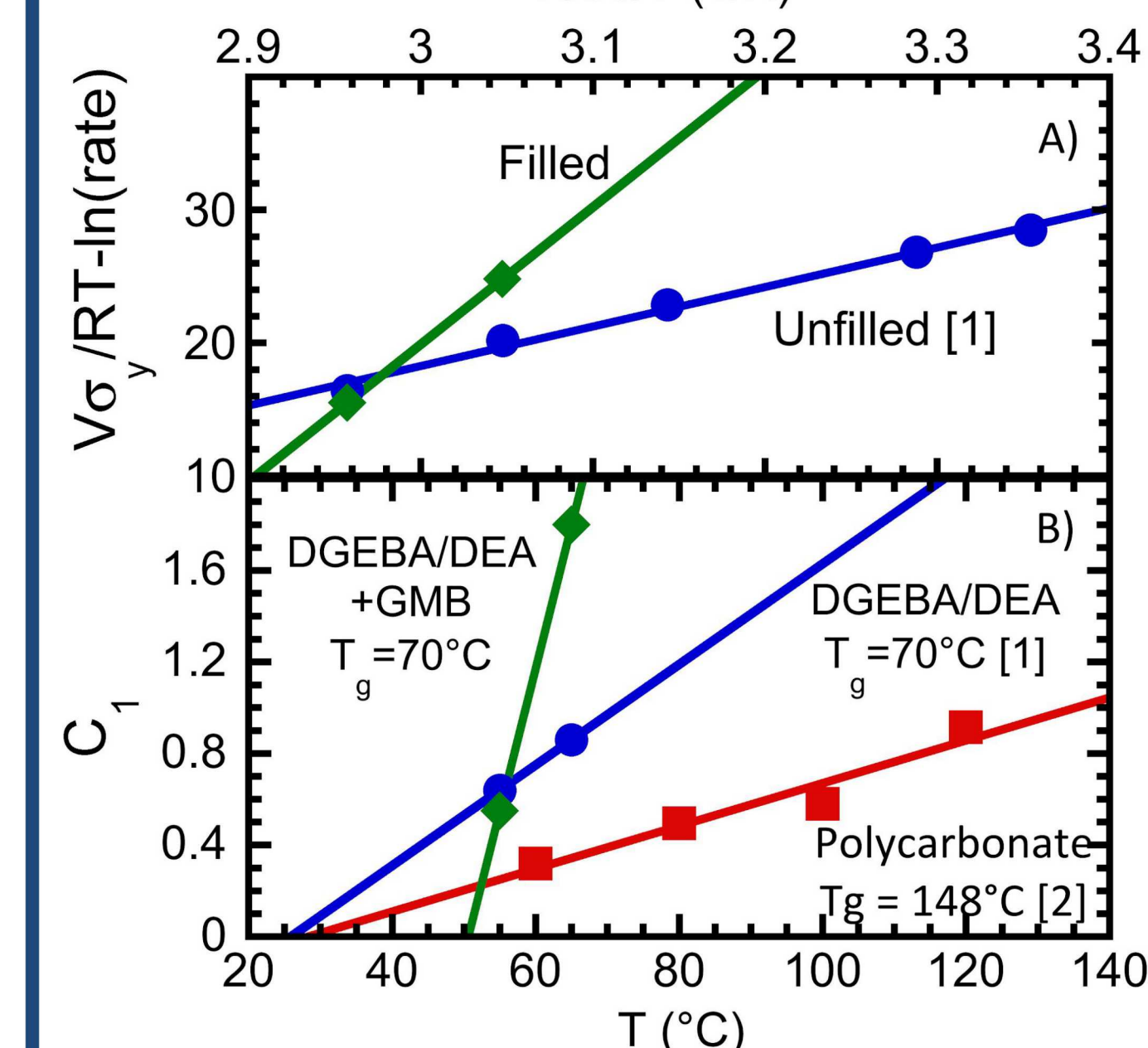
$$\sigma_Y/T = \frac{R}{V_a} \left[\frac{\Delta H_a}{RT} + \ln(2\dot{\epsilon} / \dot{\epsilon}_0) \right]; \ln(\dot{\epsilon}_0 * min) = C_2 - C_1 \ln(t_{age} / t_0).$$

Model Parameters [1, 2]

- t_0 = "Unaged" time
- V_a : Activation Vol.
- H_a : Activation Enthalpy
- C_1, C_2 : Eyring Constants
- Previously [1, 2]
 - DGEBA/DEA Unfilled
 - Polycarbonate

For $t_{age}=t_0$

$$\frac{V_a \sigma_Y}{RT} = \left[\frac{\Delta H_a}{RT} + \ln(2\dot{\epsilon}) - C_2 \right]$$

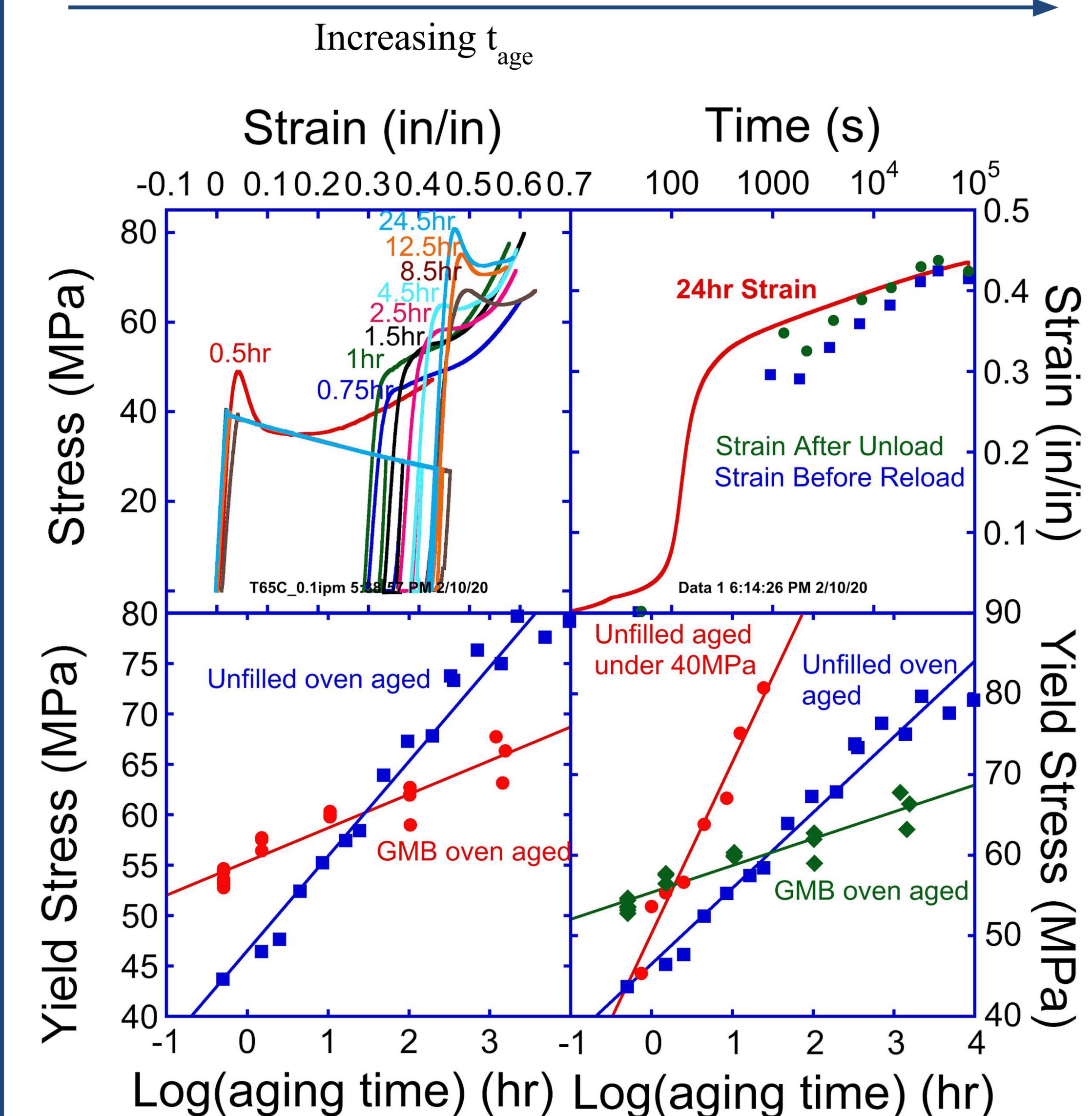


	Unfill	Fill
V_a (nm ³)	1.1	1.1-2.8
$C1(55)$	0.62	0.55
$C1(65)$	0.83	1.8
ΔH_a (kJ/mol)	268	856
$C2$	53	290

Aging Under Stress

- Unfilled 828DEA Compression cylinders
- Equilibrated at 65C for 30 minutes
- Compressed at a rate of 0.1ipm to a stress of 40MPa
- Held at 40MPa for $t_{age} = 0.25hr$ up to 24.5hr
- Unloaded until crosshead displacement was 0 in.
- Compressed at 0.1ipm to yield

Permanent Set (after full testing sequence)



Summary

- Epoxy samples of 828DEAGMB have been compression tested over a range of temperatures and aging times
- Data used to find parameters from the Ree-Eyring Theory
- Results then compared to unfilled system
- Yield stress peak evolution found to accelerate when aged under stress
- Aging rates: Unfilled under stress > Unfilled > Filled

References

- [1] Arechederra, G. K., et al. (2019). "Interplay between chemical and physical aging in diethanolamine cured diglycidyl ether of bisphenol-A (DGEBA) epoxy." Elsevier.
- [2] C. H. Huu, T. Vu-Khanh, (2003) "Effects of physical aging on yielding kinetics of polycarbonate", Theor. Appl. Fract. Mech. 40(1) 75-83