Defence Academy of the United Kingdom

Cranfield UNIVERSITY

Whole Life Assessment of Nitrocellulose in Double Base Propellants

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Contents

- Introduction
- Aged DB rocket propellant analysis
  - GPC
  - HPLC
- Aged DB gun propellant analysis
  - GPC
  - microhardness
- Conclusion and future work

The problem

Whole life assessment of NC in propellants

- Processing parameter during manufacture
  - viscosity…
- Chemical ageing
  - stabiliser depletion
  - NC molar mass changes
- Mechanical ageing
  - hardness…

GPC method described in STANAG 4178

- Repeatable results ⟹ once a month (5 months)

same batch of NC was analysed in 20 replicates

	RSD (Mn)	RSD (Mw)
5 measurements	2.8%	2.8%

- RSD 10-100% in the past
- Comparable to other analytical techniques

Validation of the GPC method

- Broad distributed PS standard NBS 706a
- Certified by NIST (previously NBS)
- Injected in triplicate with every set of propellant samples

Accuracy and precision of the GPC method

14 measurements of NBS 706a Mw in triplicate (5 month)

- Accuracy:
  - Mw = 2.85 ± 0.23 10^5 g/mol (± 8%) certified by NIST
- Precision: RSD < 0.9%

The aim

- To assess the life of DB propellants by:
  - transferring the GPC method to formulated NC product (aged DBP)
  - measuring the kinetic parameters (k, E_a)
- To perform mechanical testing and correlate results to average molar mass of NC

Propellants used

- **Generic DB rocket propellant**
  - NG, NC, p-NMA and 2-NDPA
  - supplied by Roxel (UK Rocket Motors) Ltd
- **DB gun propellant, 20 mm (Phalanx)**
  - NC, NG, DPA
  - donated by DOSG MoD
  - in addition naturally aged propellant (15 y old) was supplied

Ageing (AOP-48)

2g for DB rocket propellant / 5g for DB gun propellant were

- pre-conditioned
  - 60-70% RH level, 48h, 25^{o}C
- heat sealed in polymer coated aluminium bags
- aged at
  - 30, 40, 50, 60^{o}C (DB rocket propellant)
  - 70, 80^{o}C (DB gun propellant

GPC sample preparation

- Simple, quick and easy with no pre-conditioning
- 24h in solution
- Automatic injection in 5 replicates

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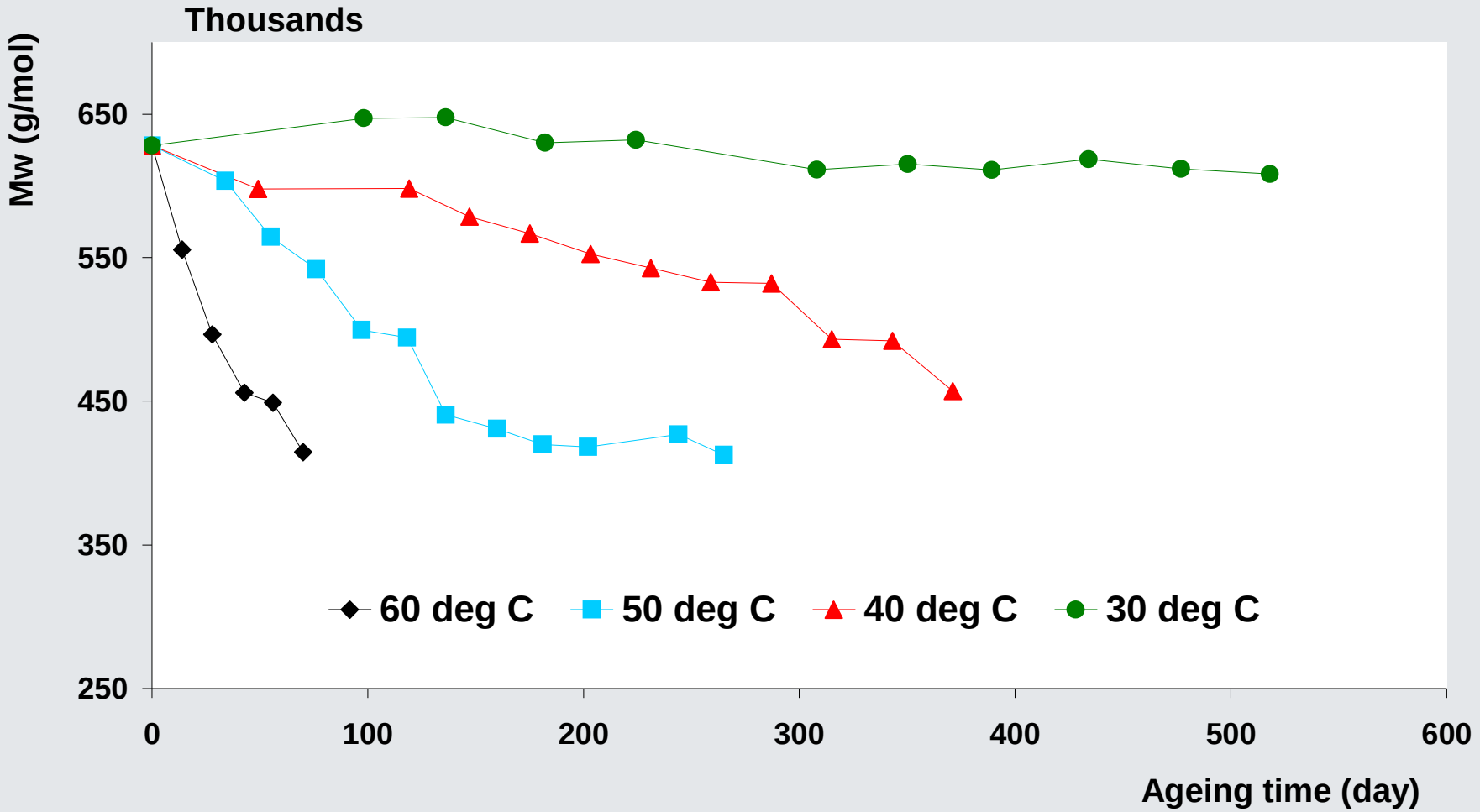
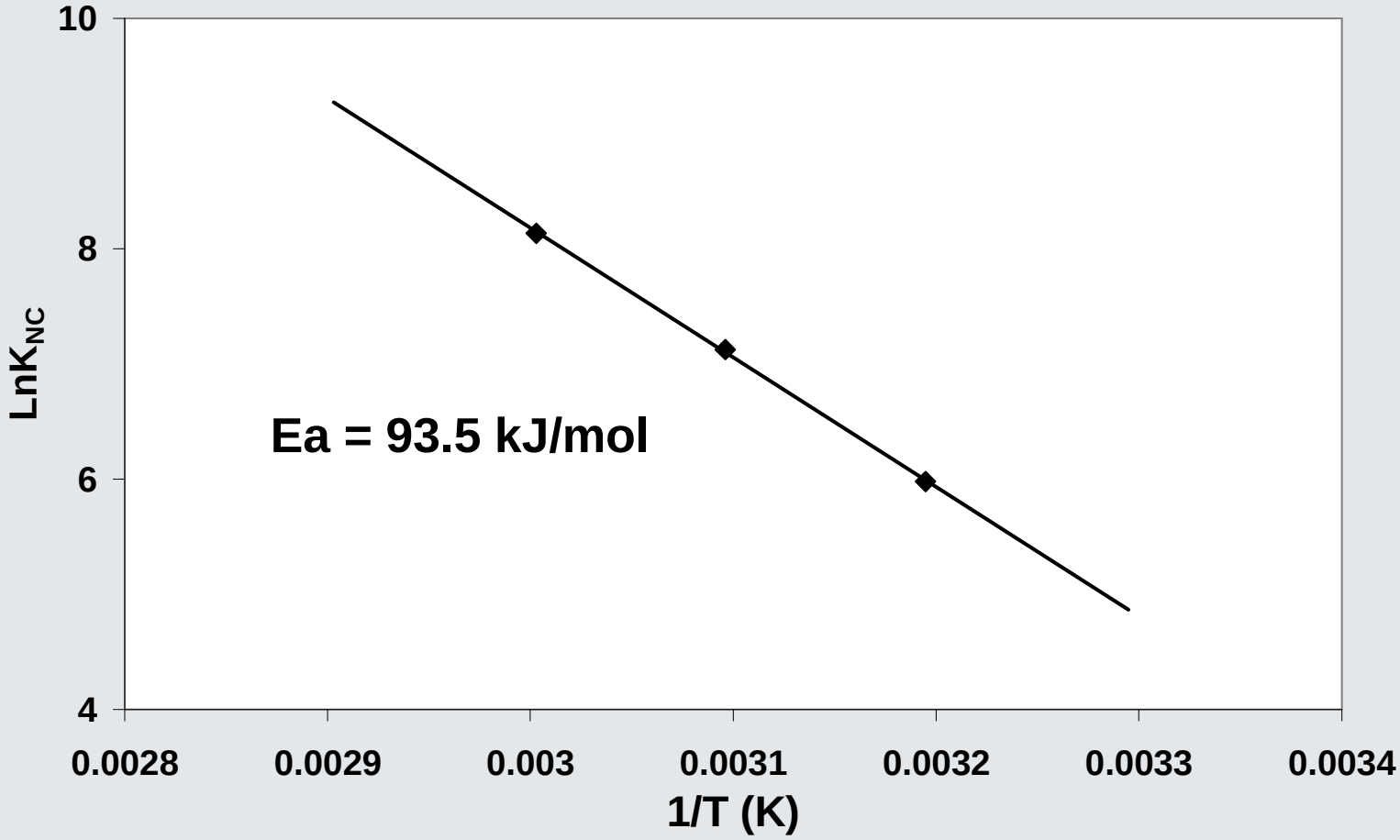
Mw changes for artificially aged DB rocket propellant



- Mn follows the same trend

Arrhenius plot for NC decomposition



Decomposition of NC at T<60^{o}C dominated by low Ea process.

NC decomposition

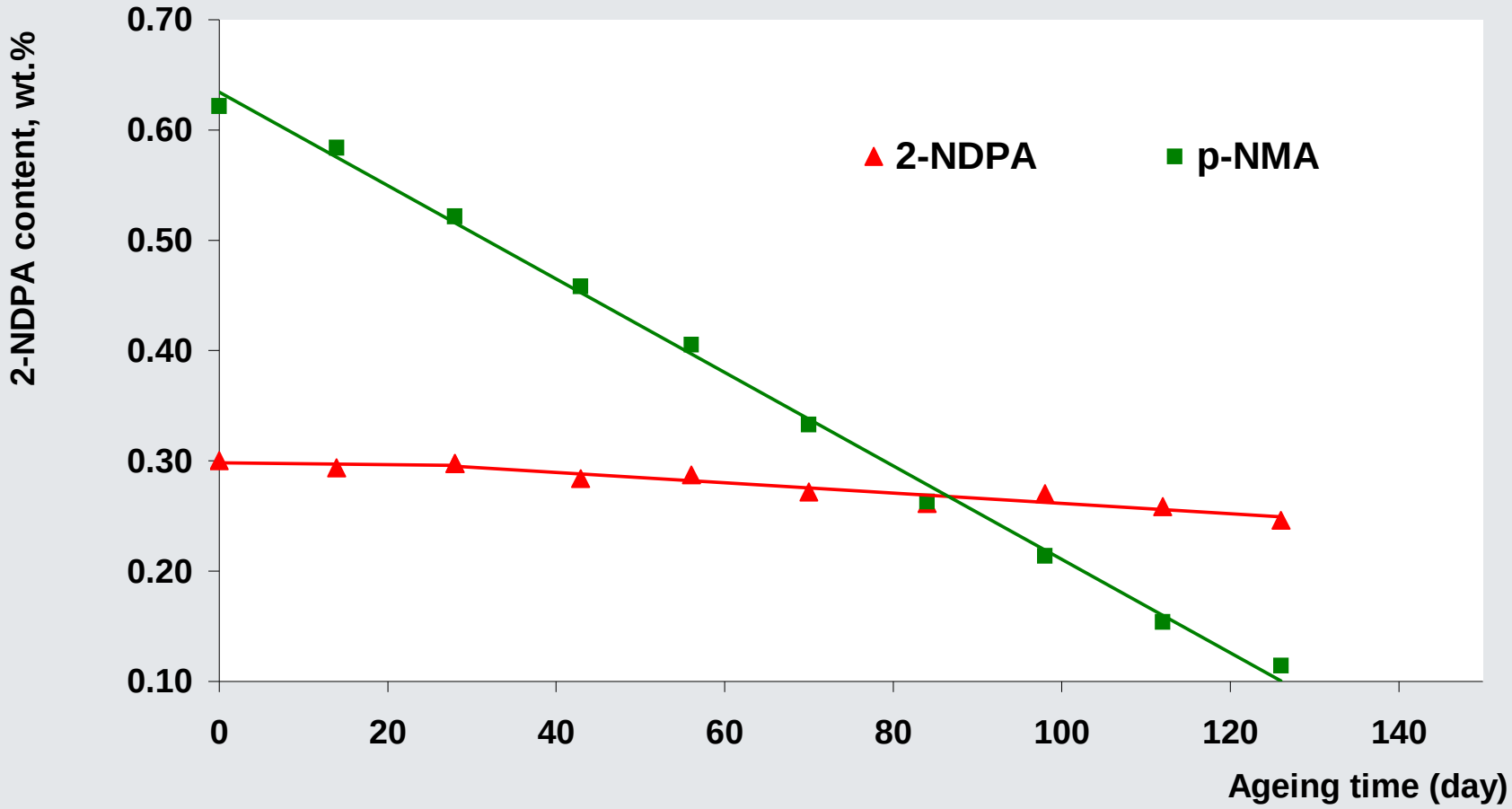
NC decomposes following 2 pathways:

- Thermolysis of CO-NO_2 group (producing 2 radicals) Ea = 160-170 kJ/mol
- Hydrolysis of CO-NO_2 group (producing nitric acid) Ea = 100 kJ/mol

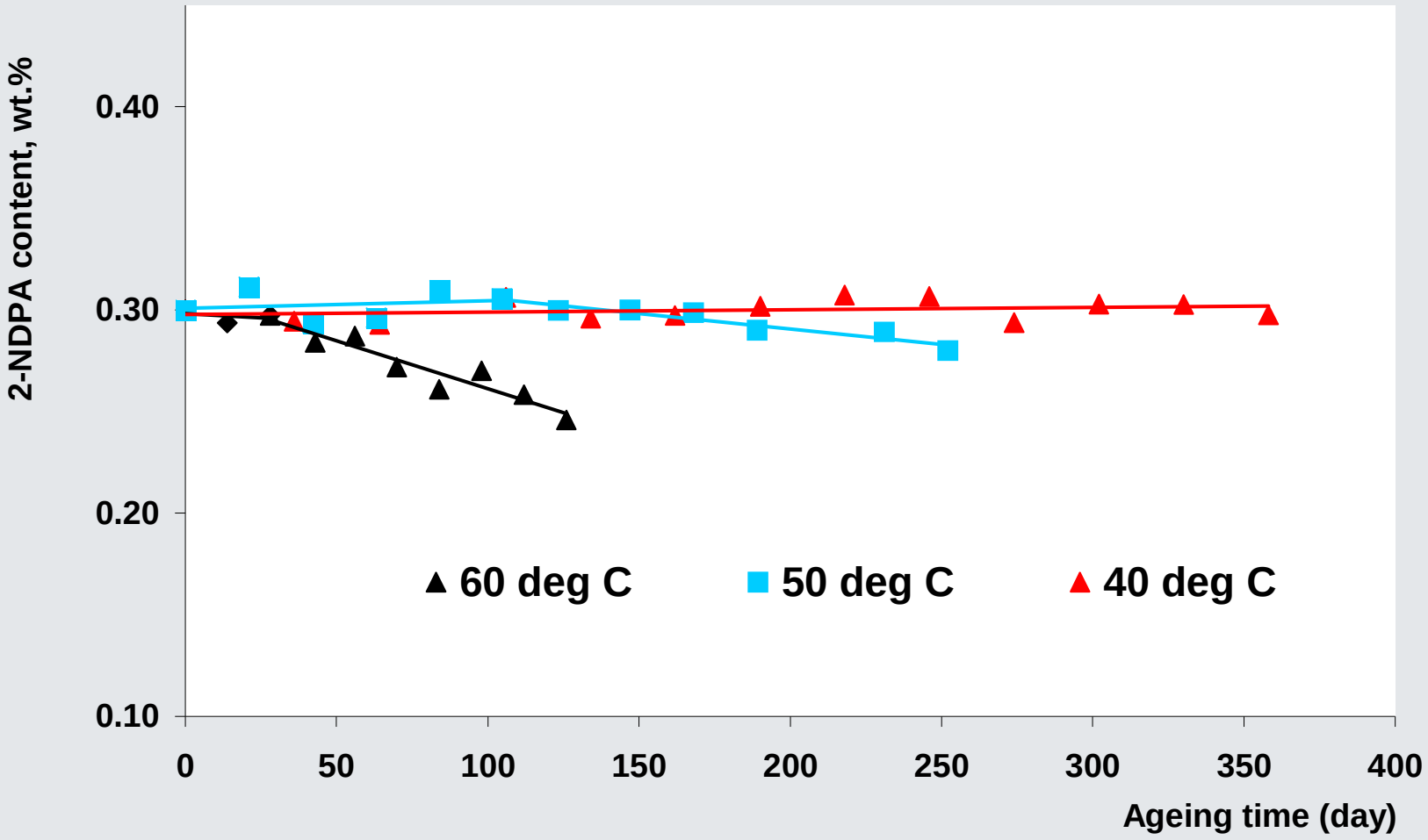
$\Longrightarrow$ Our result is consistent with hydrolysis process

Ref: M. A. Bohn, J. of Thermal Analysis and Calorimetry, 2001, 65,103

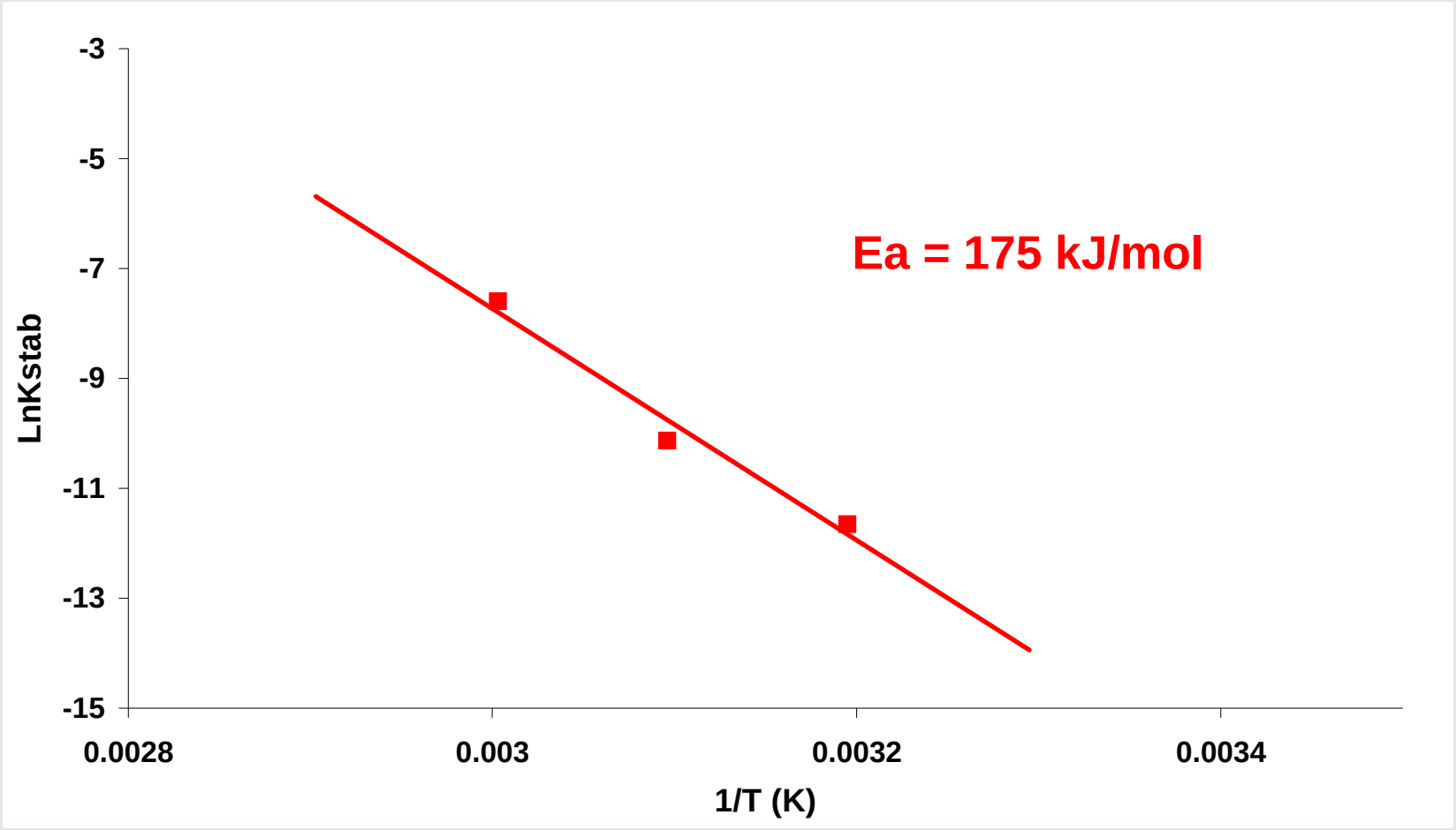
Depletion of stabilisers at 60°C



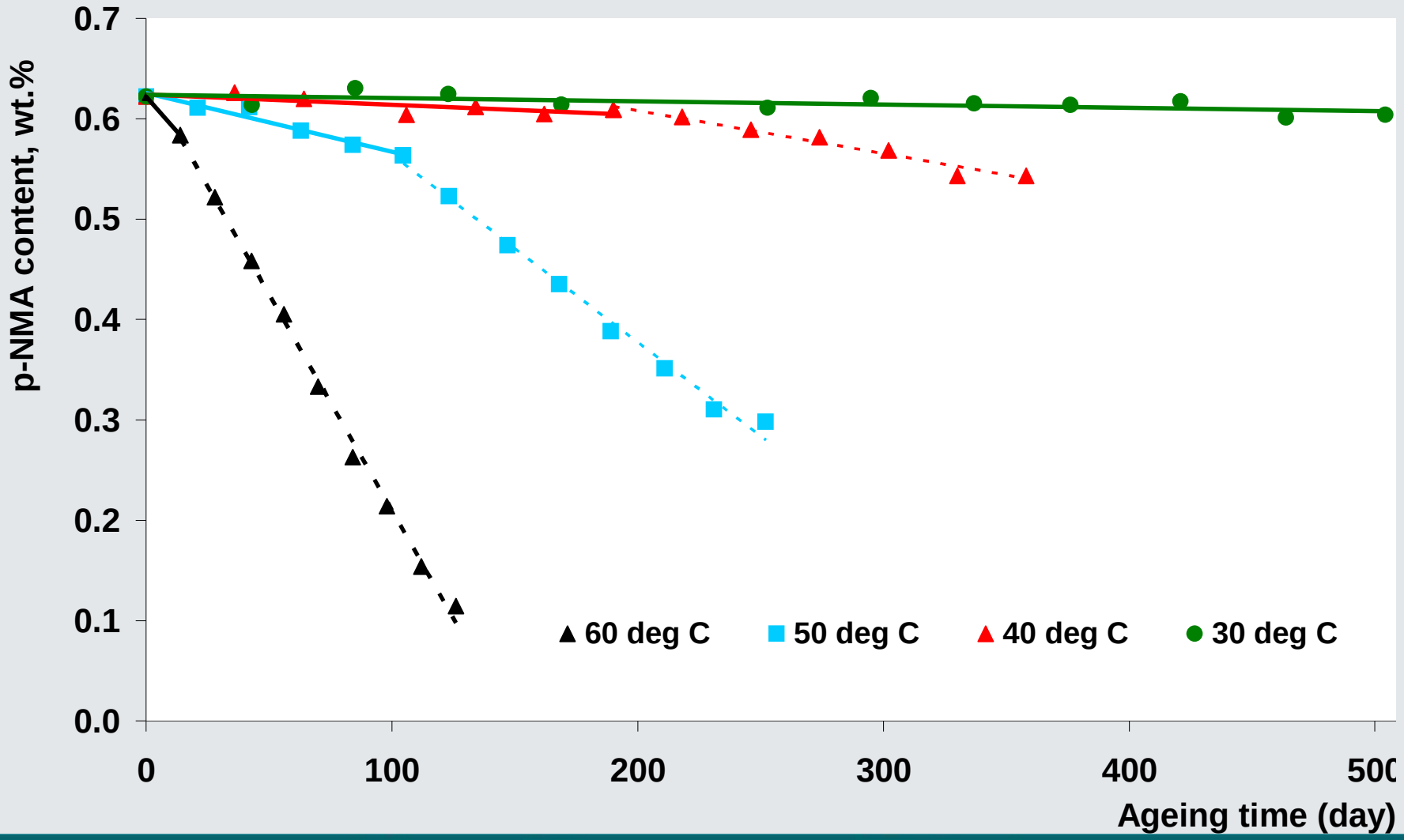
2-NDPA depletion



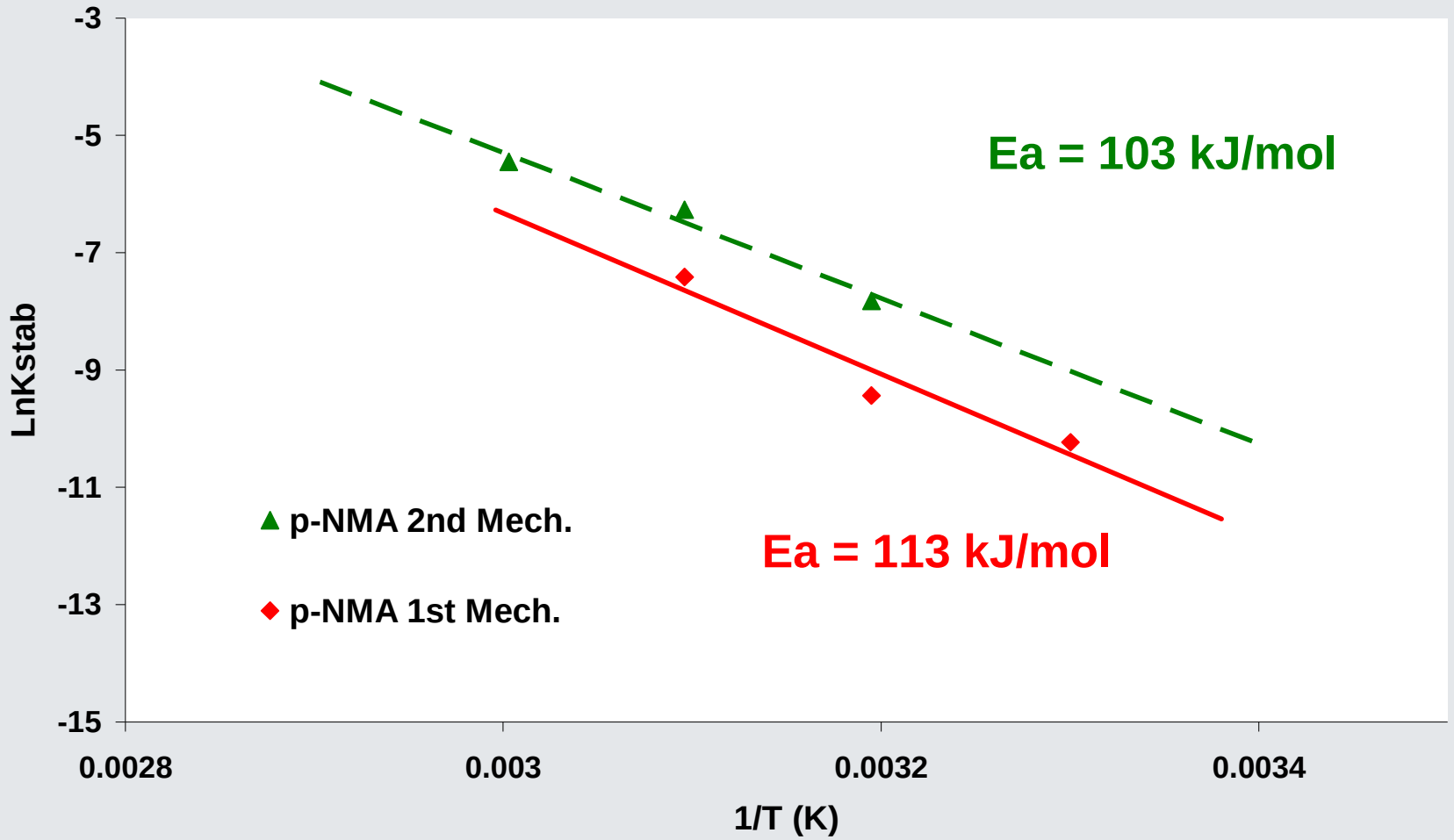
Arrhenius plot for 2-NDPA depletion



P-NMA depletion



Arrhenius plot for p-NMA depletion



Summary

- Mw and Mn decrease with ageing time
- Low Ea (Mw) consistent with hydrolysis
- p-NMA is consumed quicker than 2-NDPA with lower Ea
- 2 mechanisms involved in depletion of p-NMA
- Material too soft to do mechanical tests

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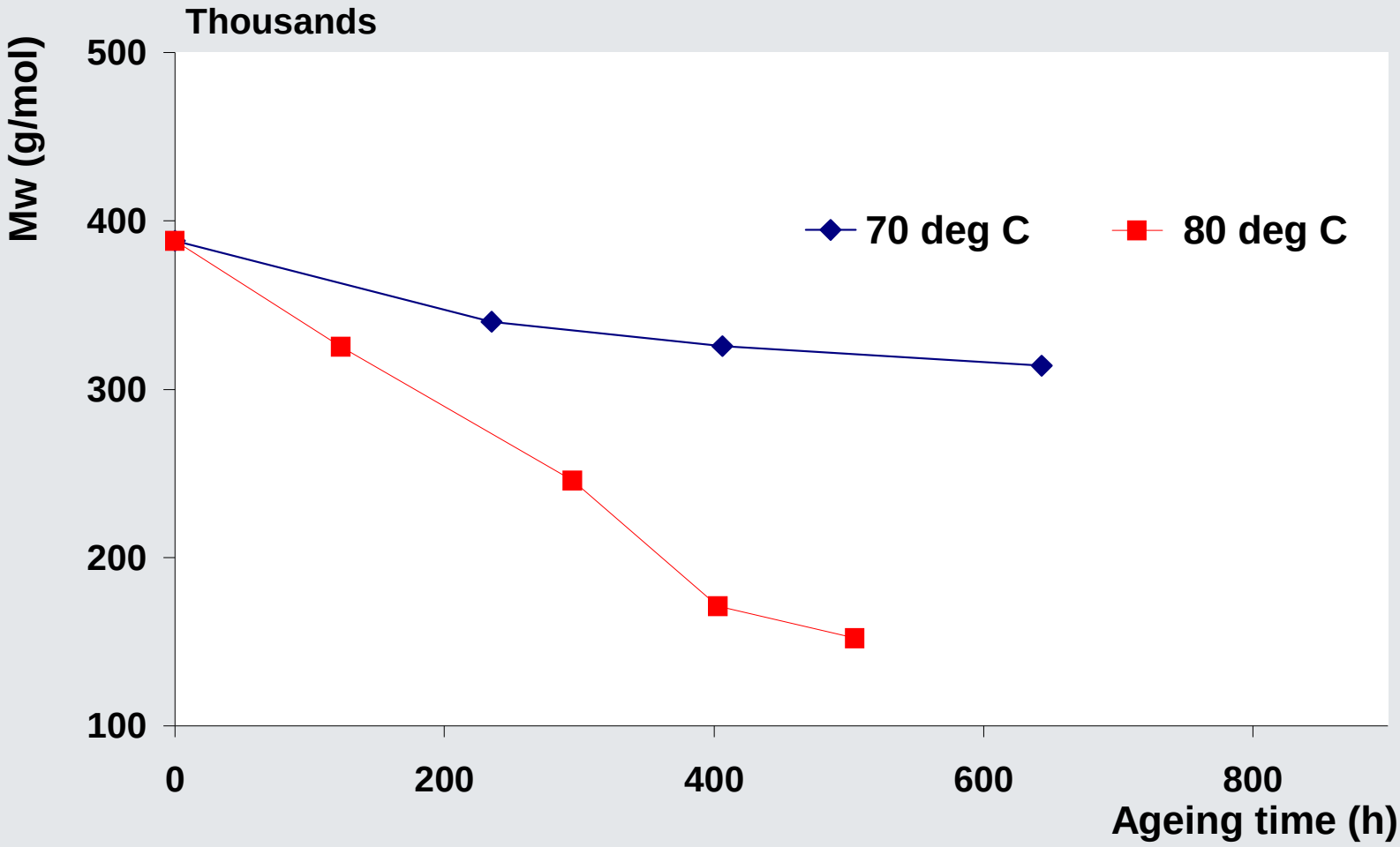
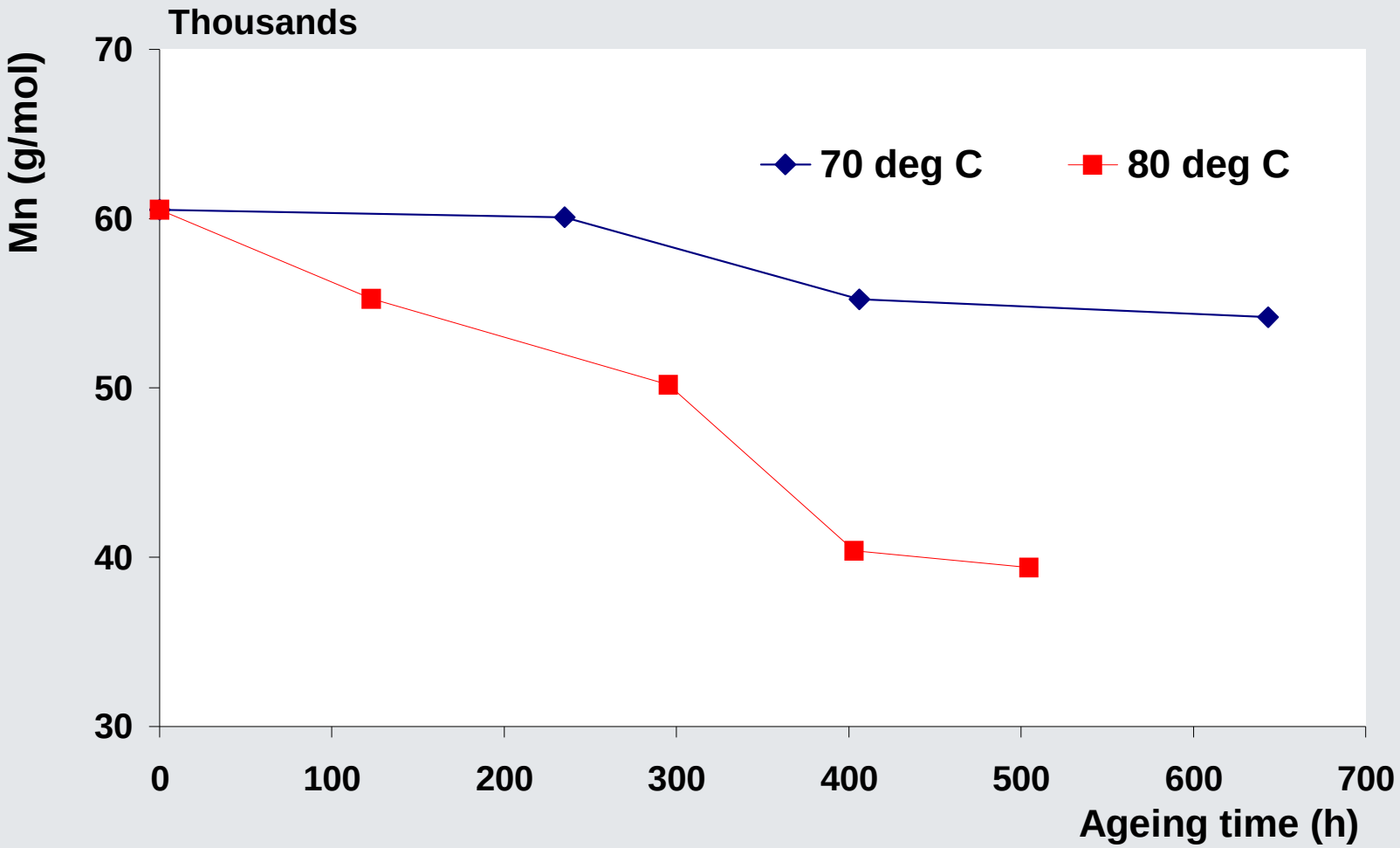
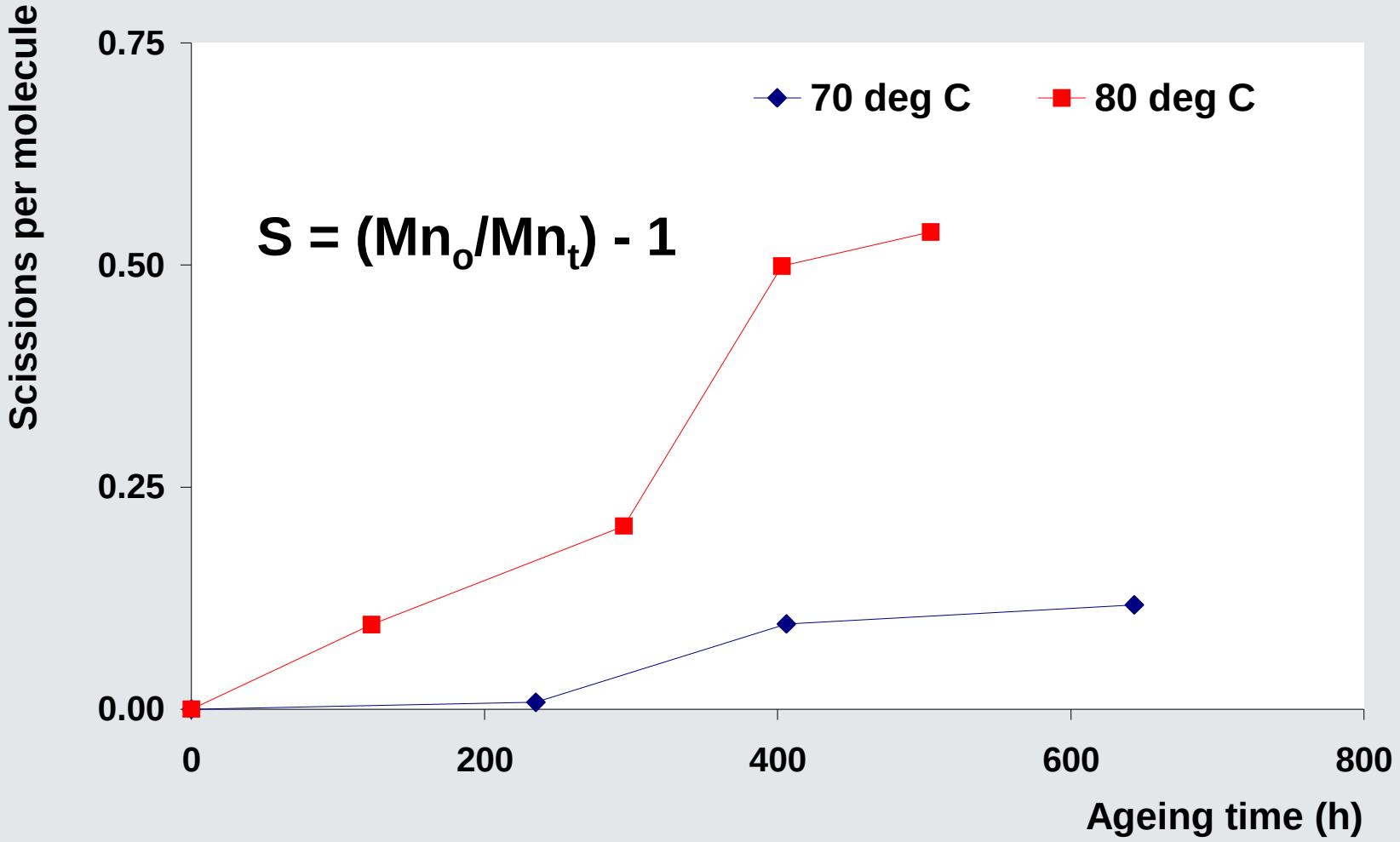
Mw changes for artificially aged DB gun propellant



Mn changes for artificially aged DB gun propellant

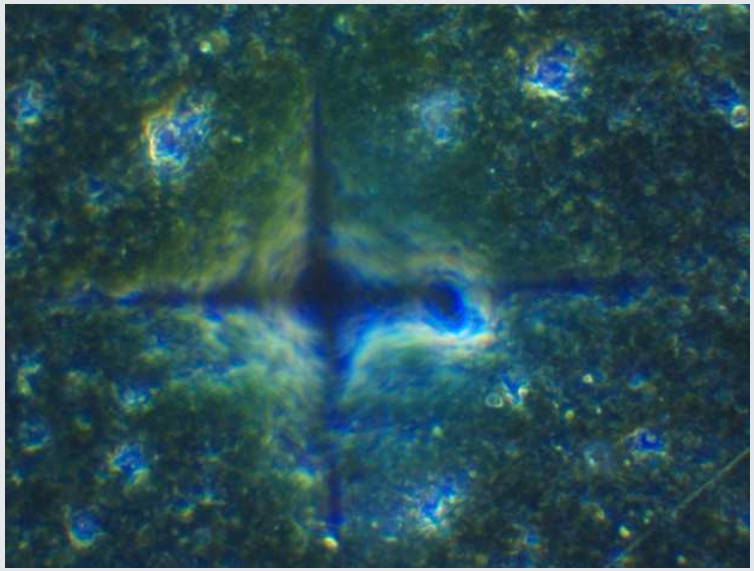


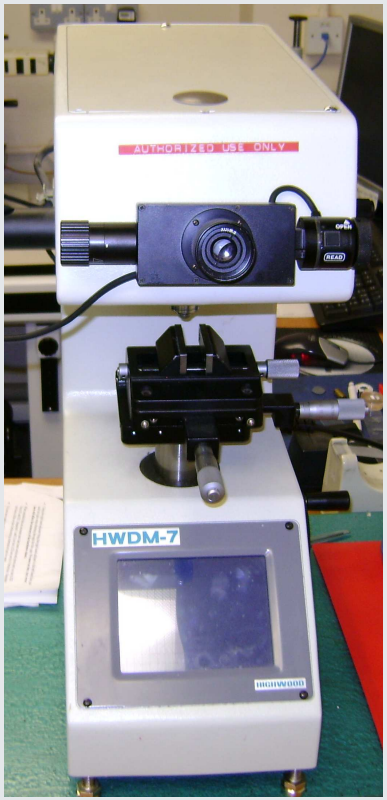
Apparent number of chain scissions of NC artificially aged at 70 and 80°C



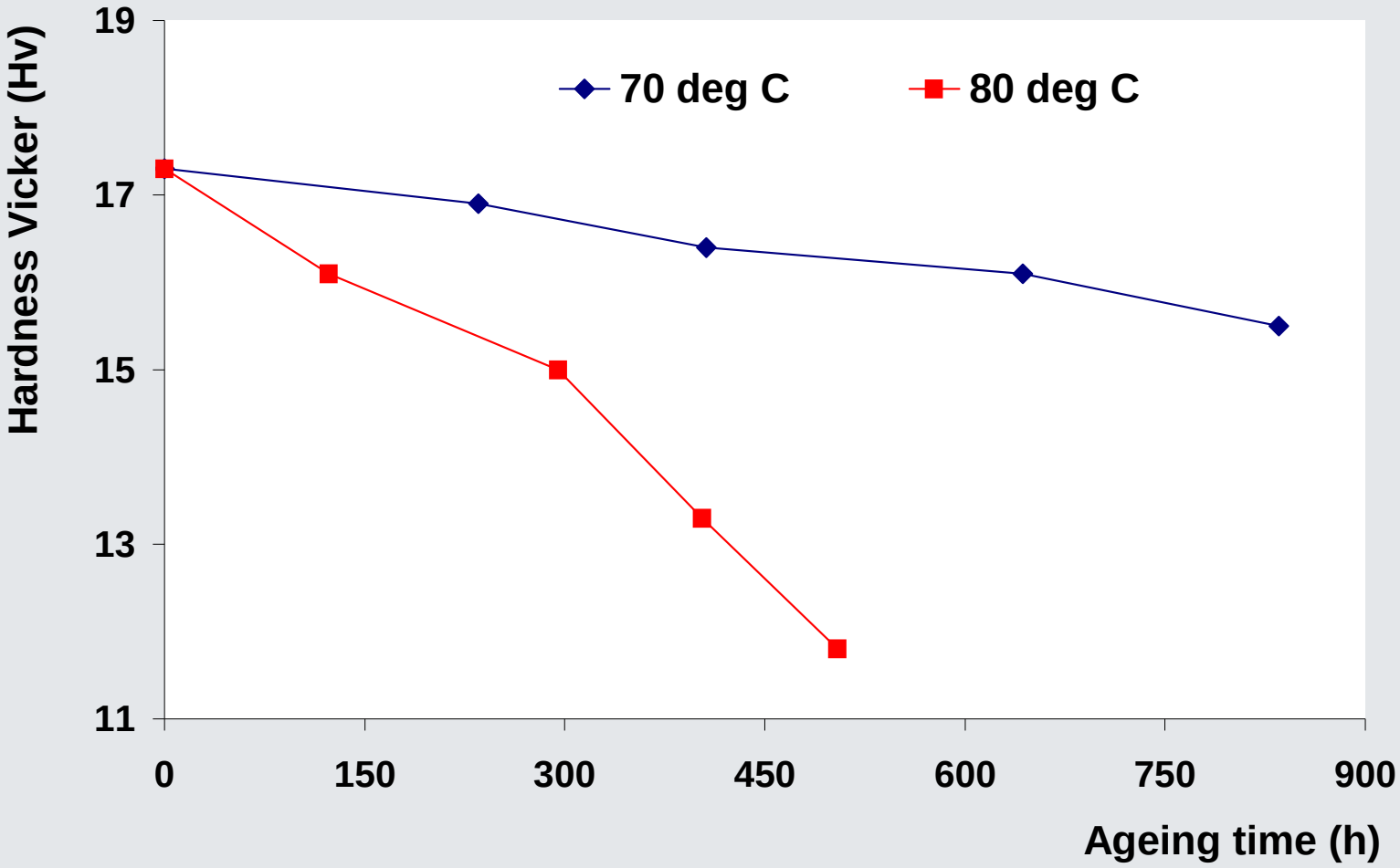
Vickers hardness test

- Easy and quick testing procedure which consists of measuring the diagonals of the impression left by a load of various magnitudes

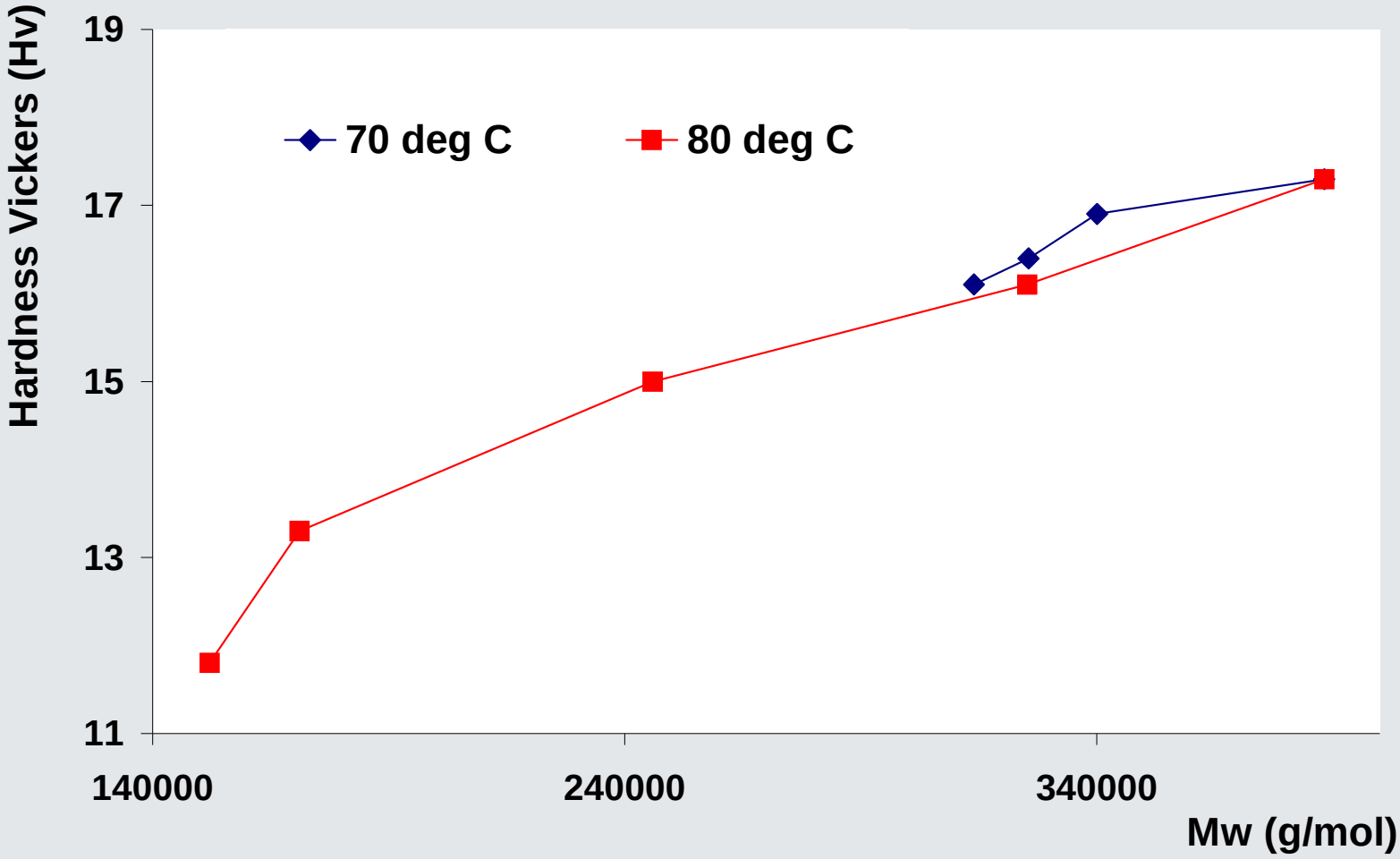




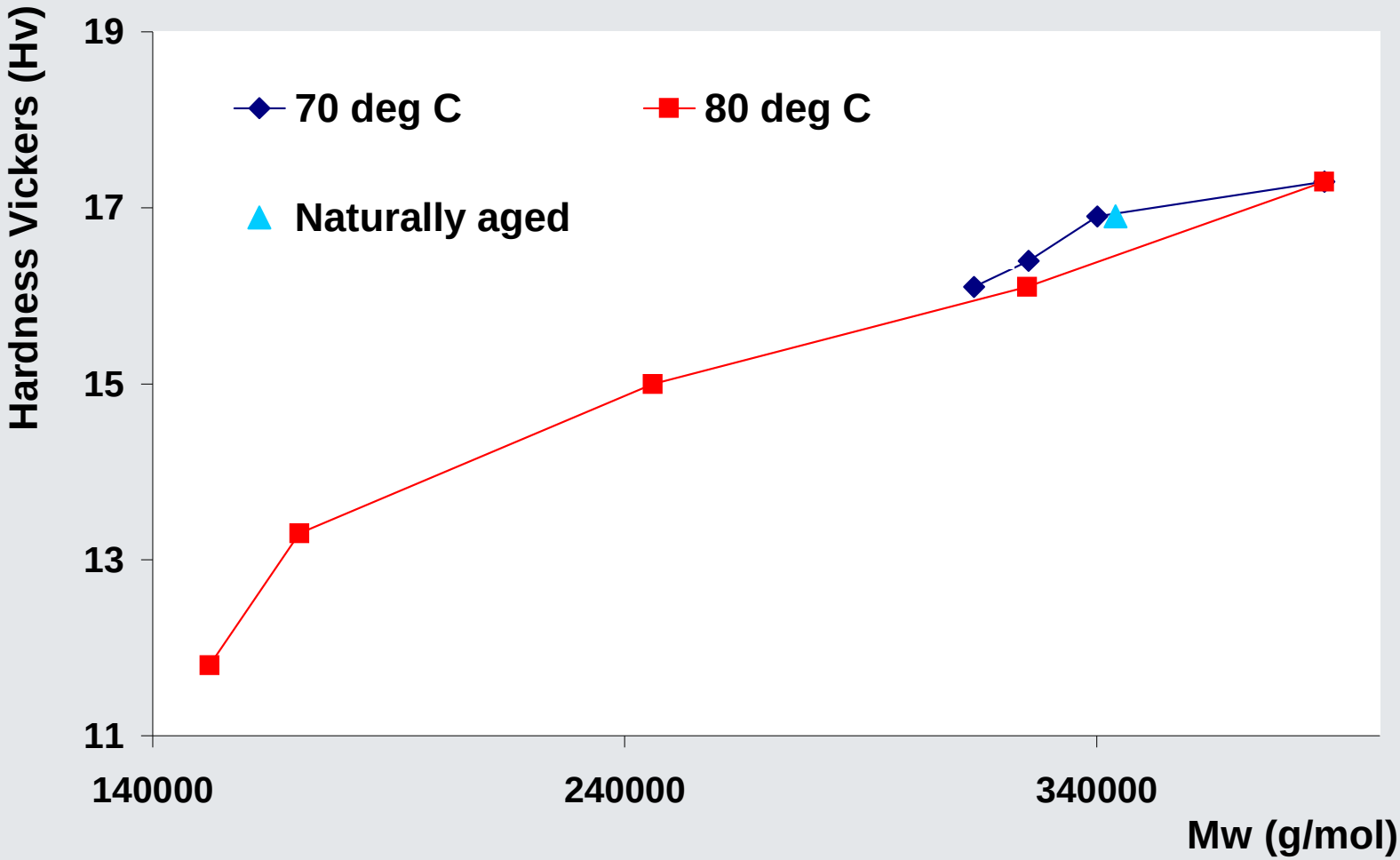
Grain hardness changes with ageing time



Relationship between grain hardness and NC Mw



Naturally aged propellant 15-16y



Summary

- Mw and Mn decrease with ageing time
- Chain scission factor consistent with hydrolysis
- Grain hardness decreases with ageing time
- Good correlation between grain hardness and Mw
- Naturally aged material fits with the curve

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Conclusion

- The GPC method successfully transferred to formulated NC
- Ea (Mw) correlates well with published values
- Good correlation between the Mw and mechanical properties.

Ongoing experiments and future work

- Reducing sample preparation time (4h using shaker)
- Comparison GPC analysis, mechanical properties for aged propellants
  - DMA, microhardness, nanoindentation
- Round Robin analysis of NC in propellants planned for 2011/2012

Acknowledgment

- AWE and DOSG for sponsoring the work
- Roxel (UK Rocket Motors) Ltd for providing the DB rocket propellant
- Cranfield University staff and students

- Thank you for your attention

Any question?

The Defence Academy Campus