The Measurement and Interpretation of Transformation Temperatures in Nitinol

Objectives

- Develop a σ-T diagram for a commercially significant NiTi wire
- Establish terminology predictive of the mechanical performance of medical devices
- Clarify several issues pertaining to the measurement and use of transformation temperatures

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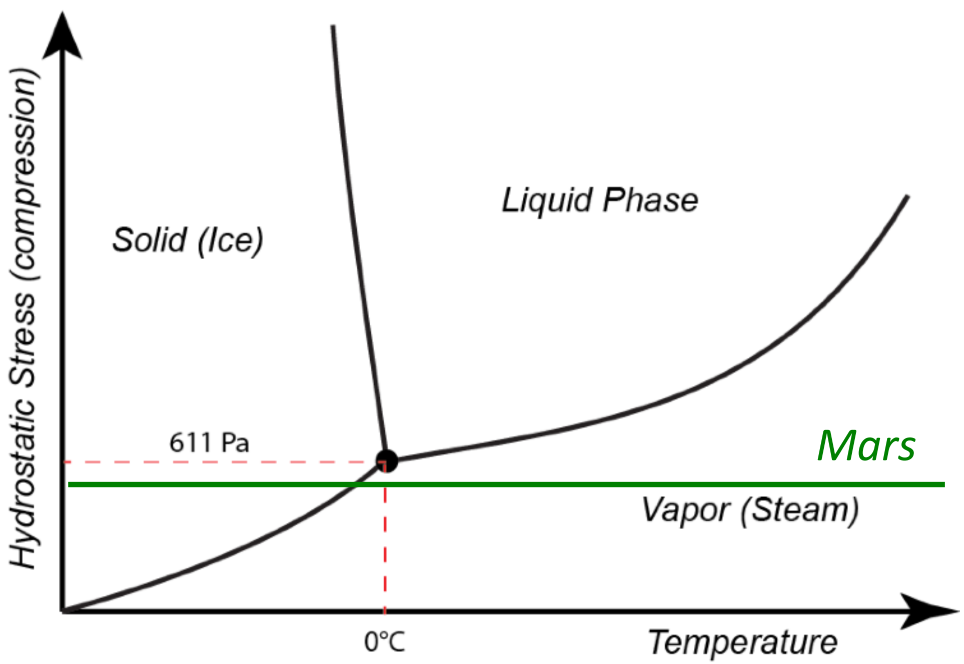
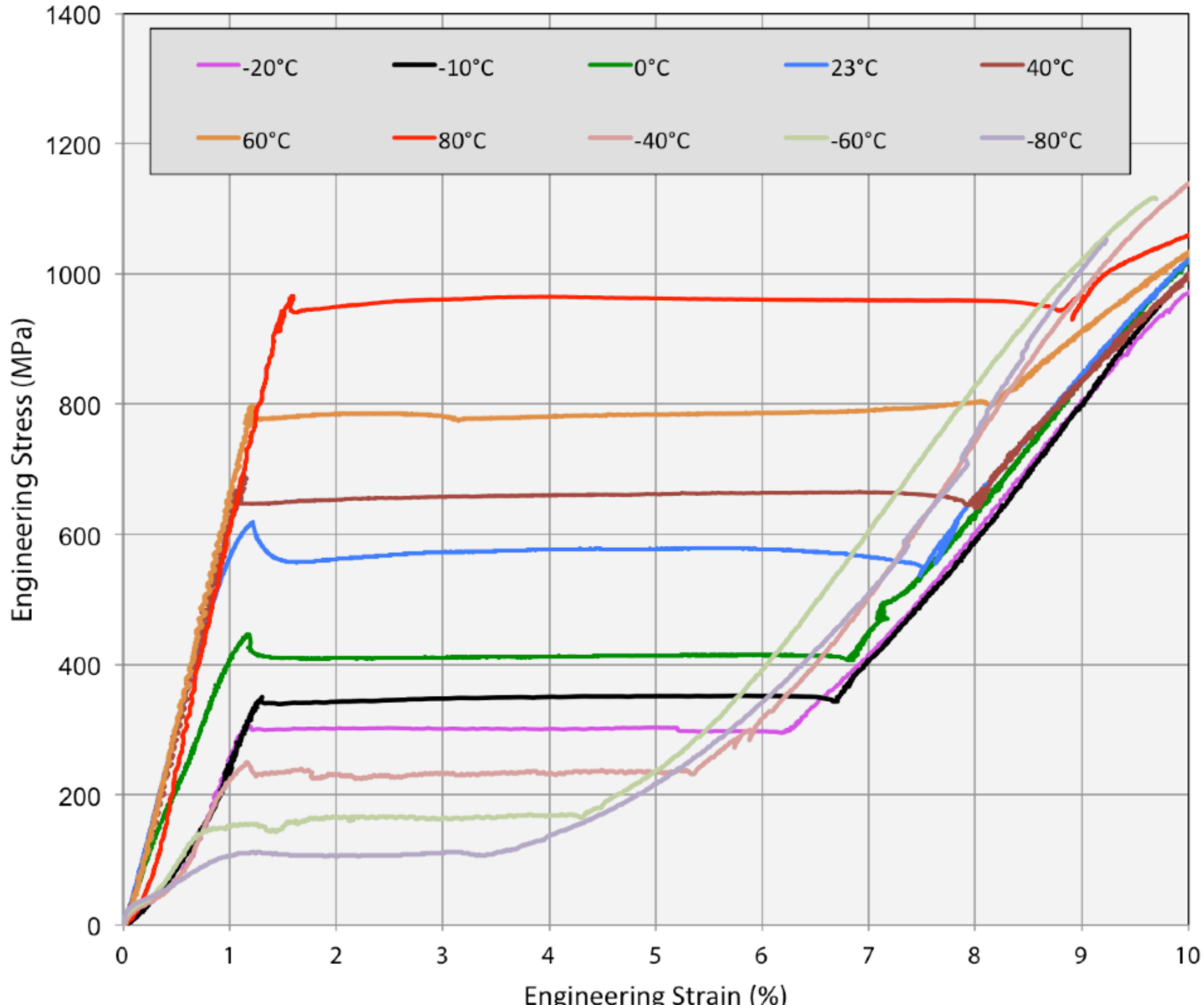
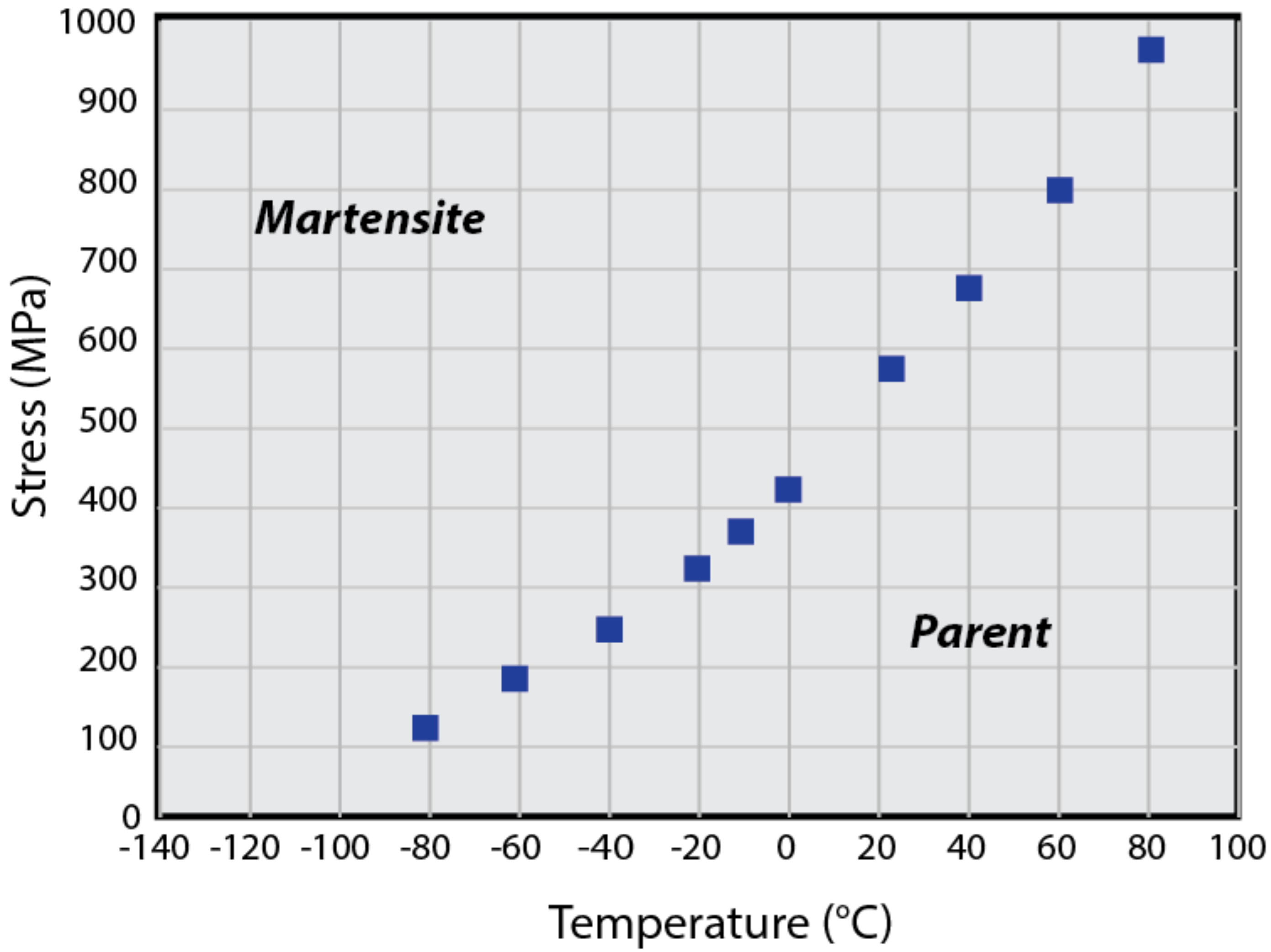


The Forward Transformation (toward Martensite) mapped by the loading plateau*

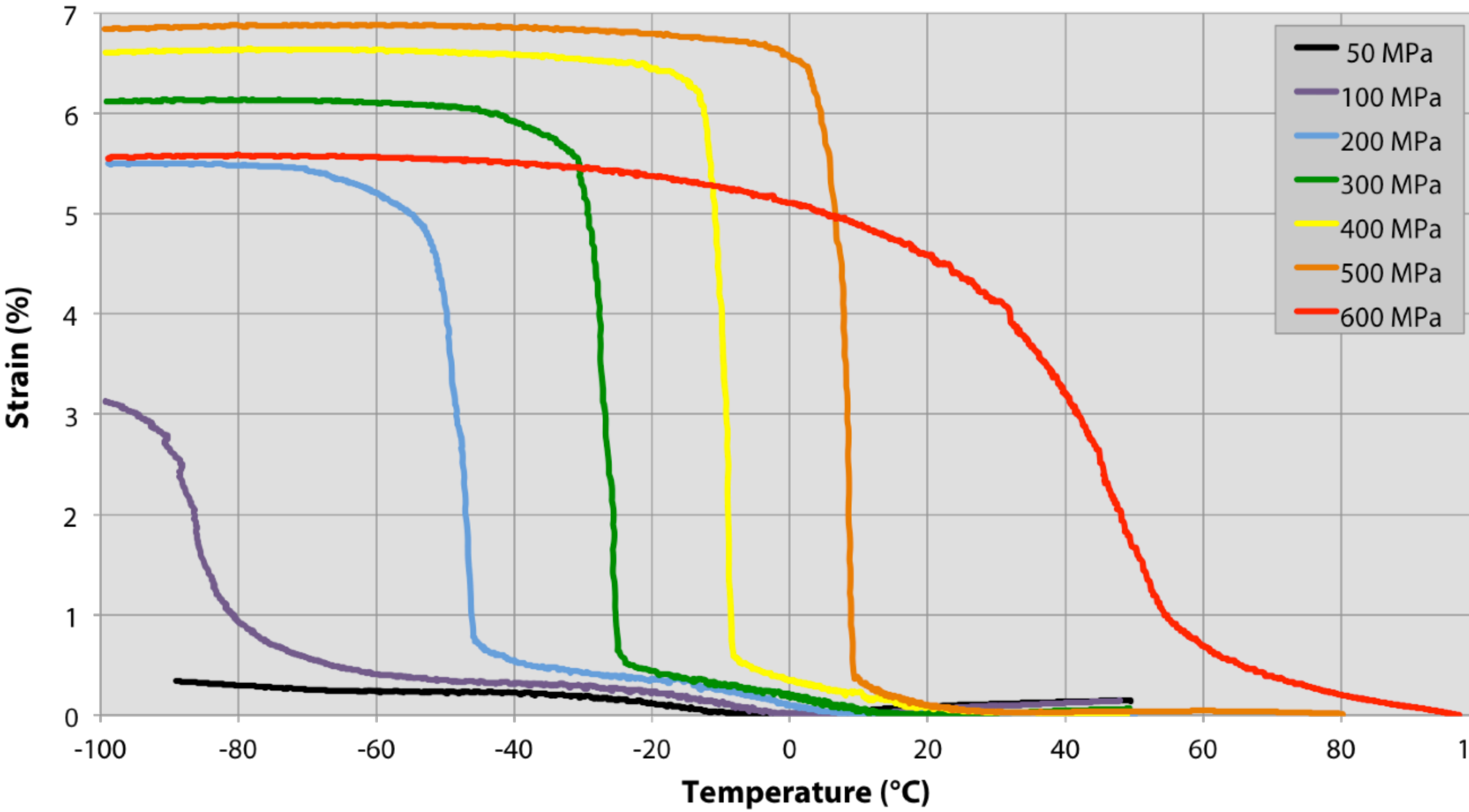


* Each test is a unique test specimen, i.e., not cyclic tests

Upper plateau stresses as a function of temperature

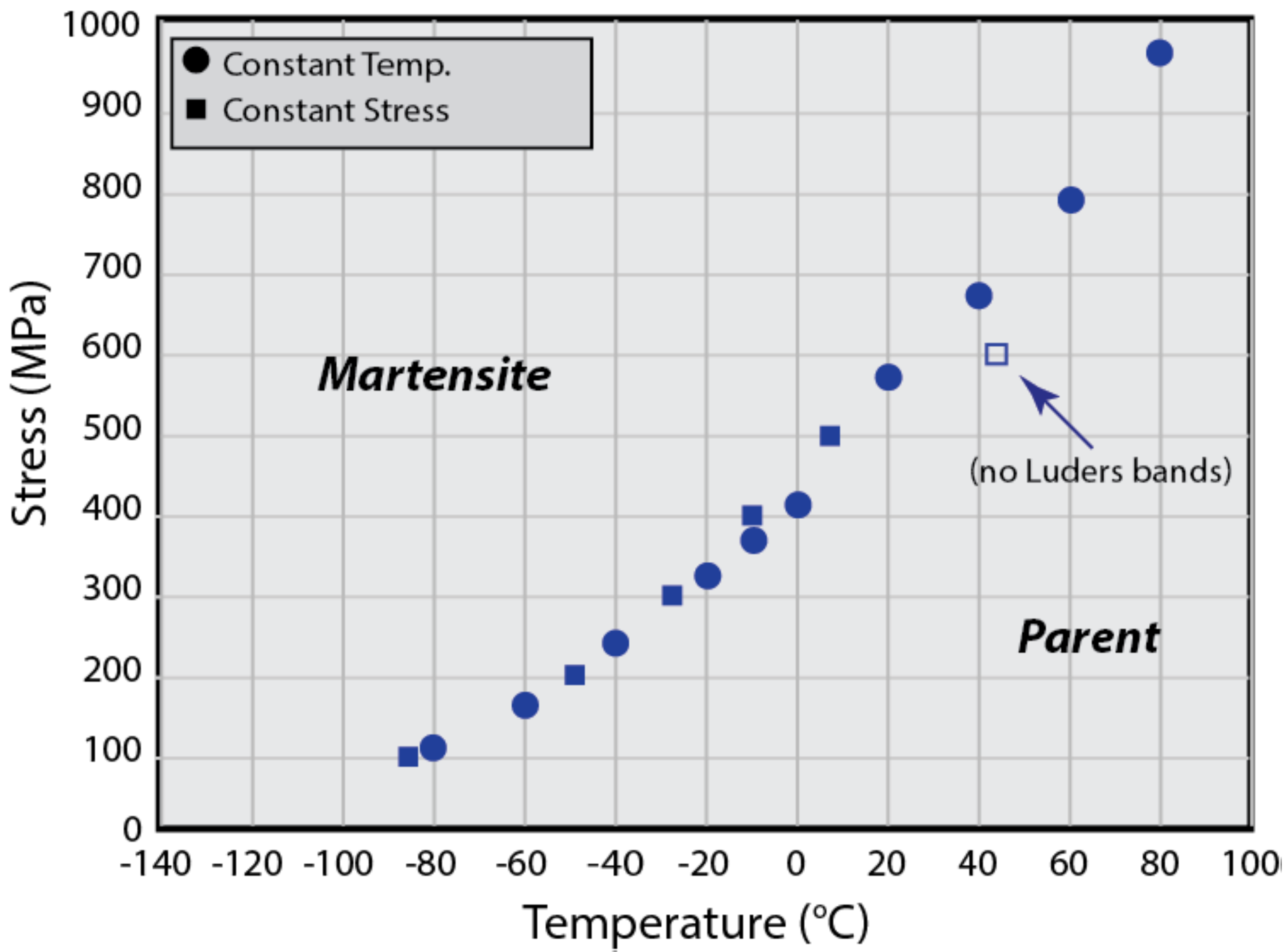


Tensile strain during cooling at constant stresses*

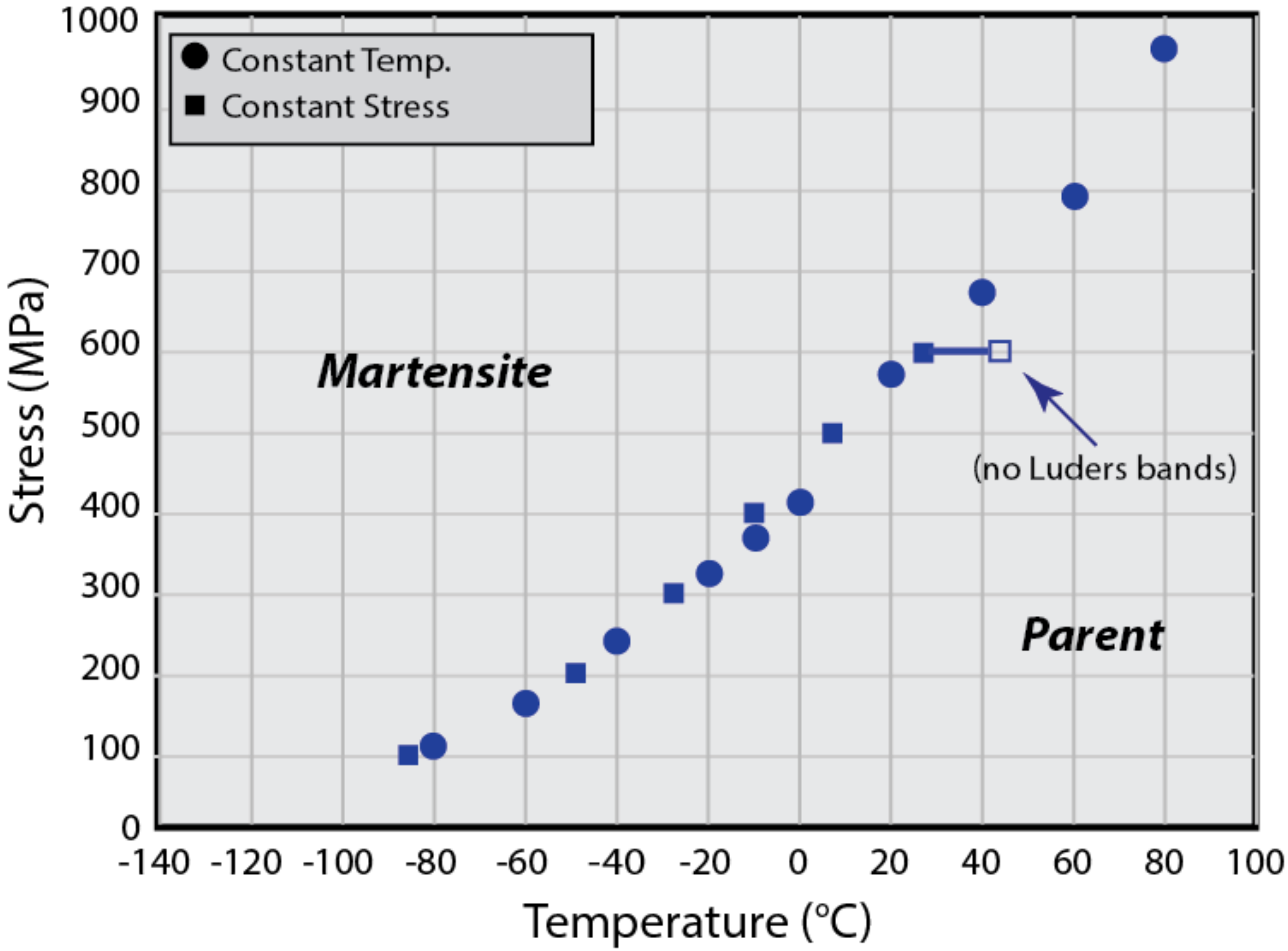


* run with video extensometer in an load-controlled, screw-based testing machine

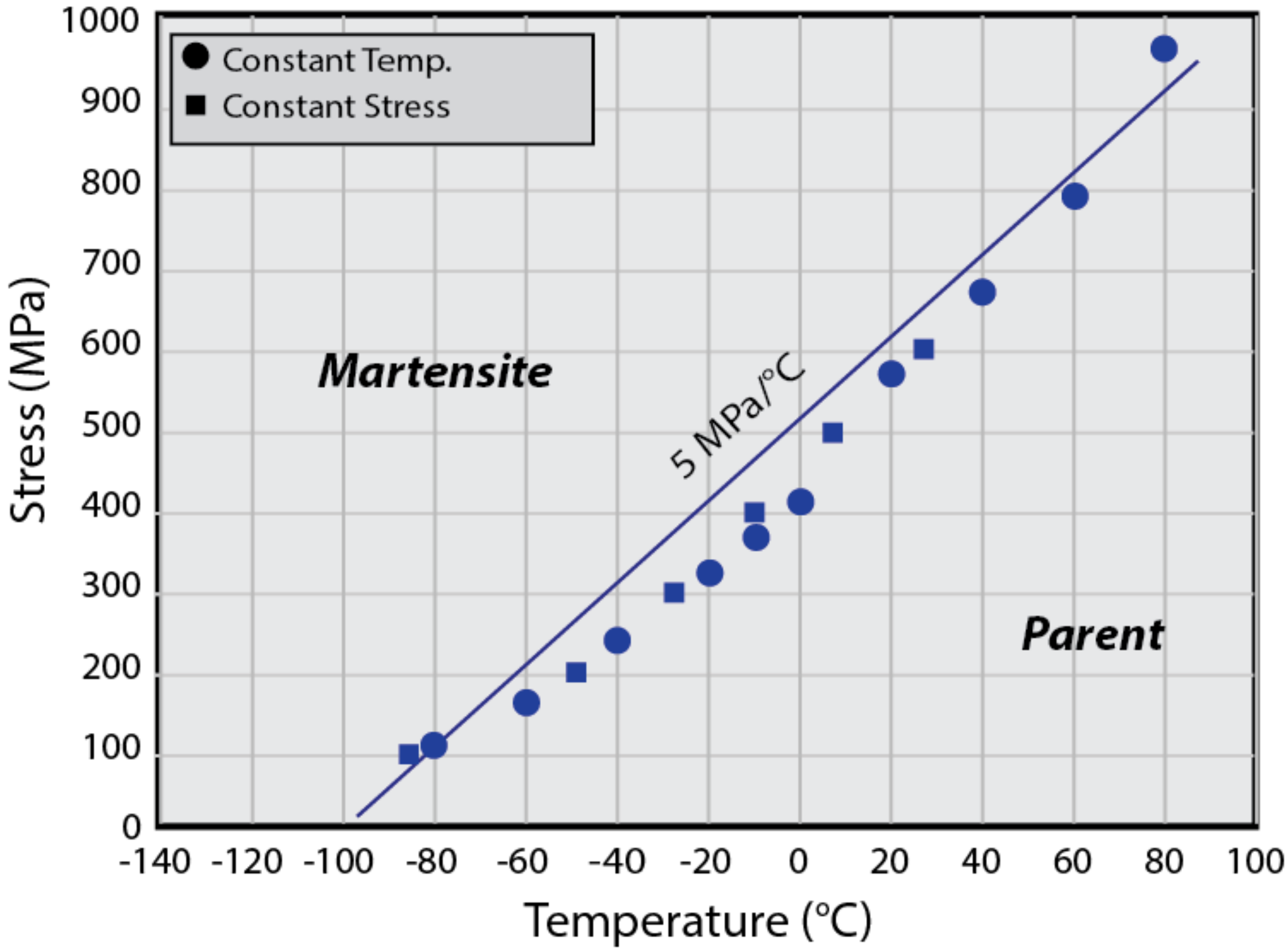
Forward transformation to Martensite



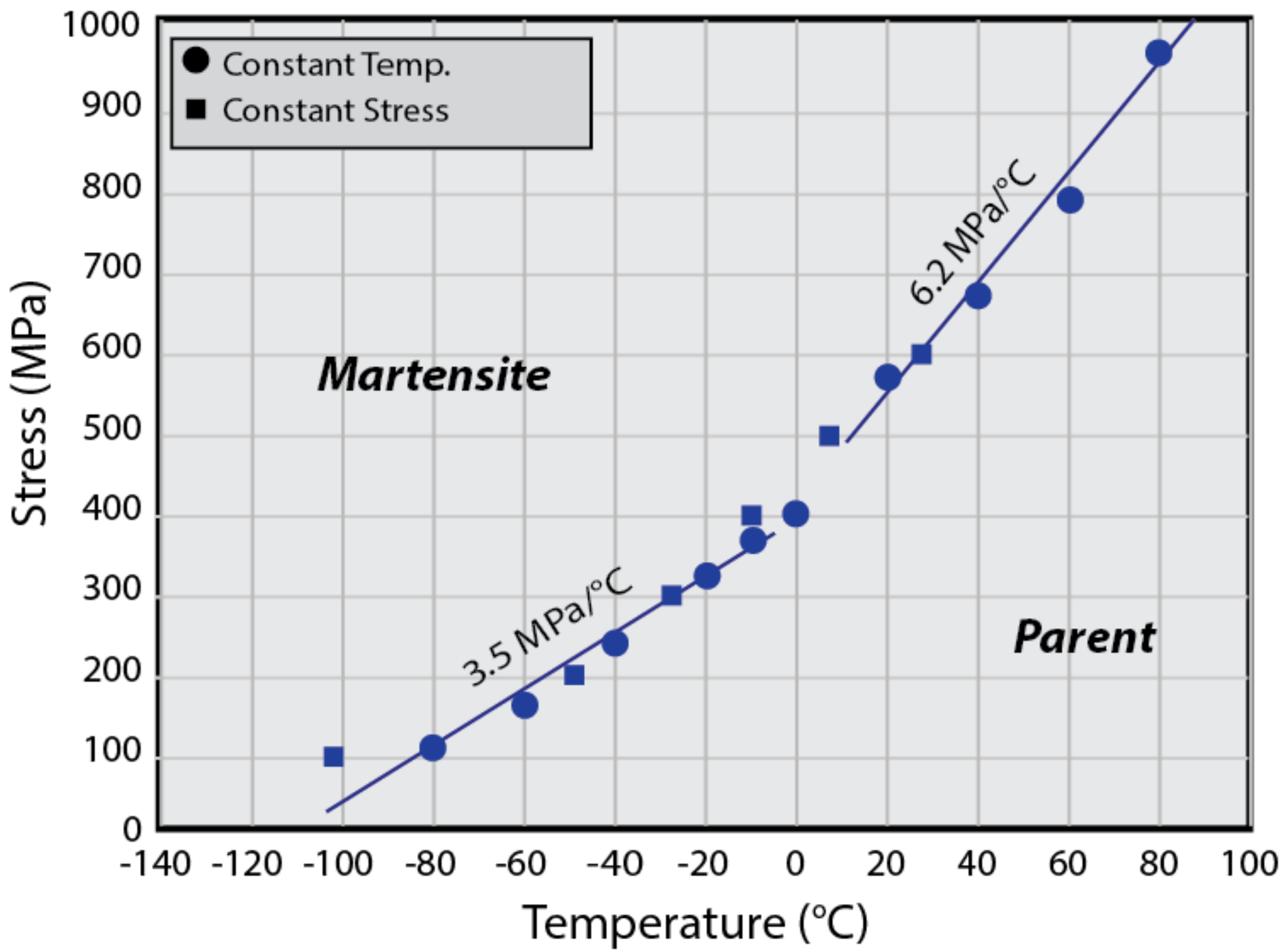
Forward transformation to Martensite



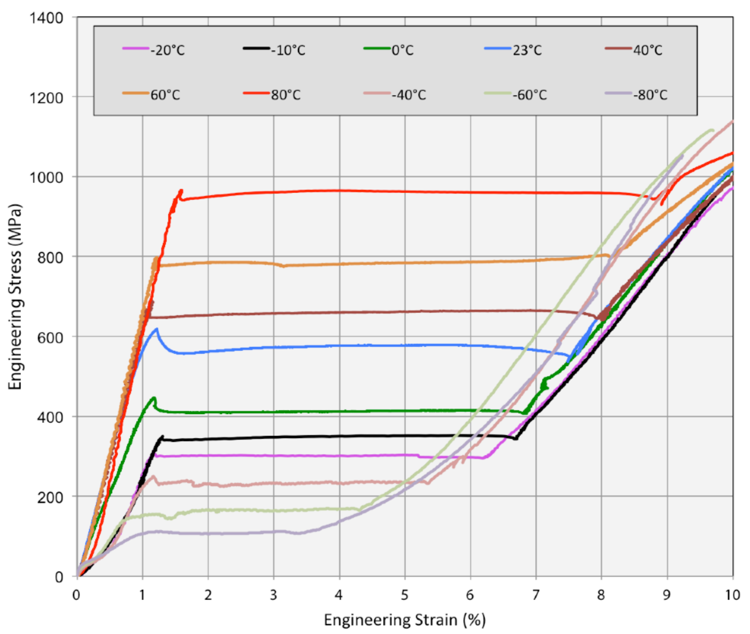
Forward transformation to Martensite

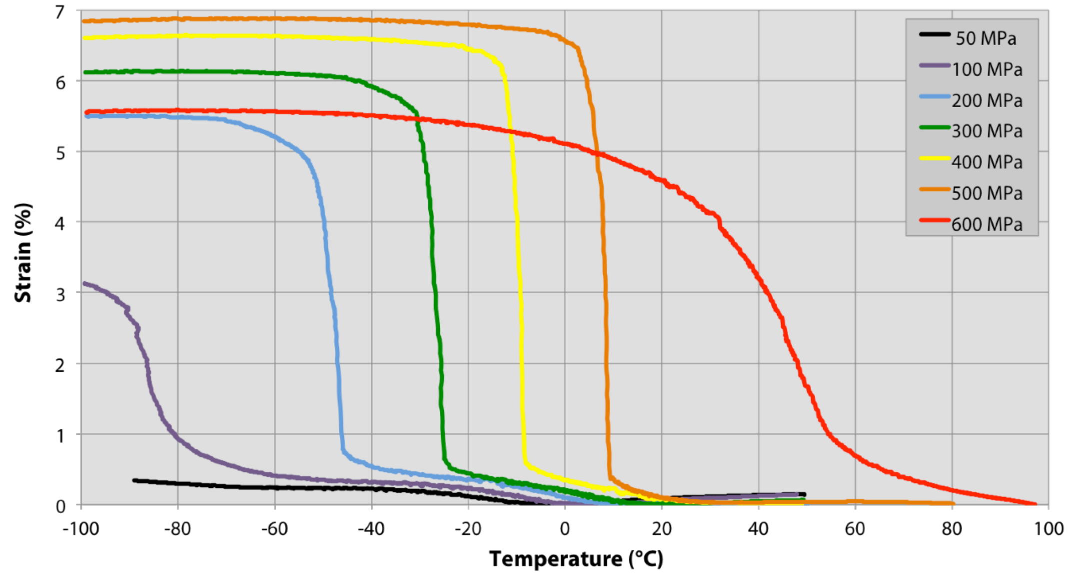


Forward Martensite transus

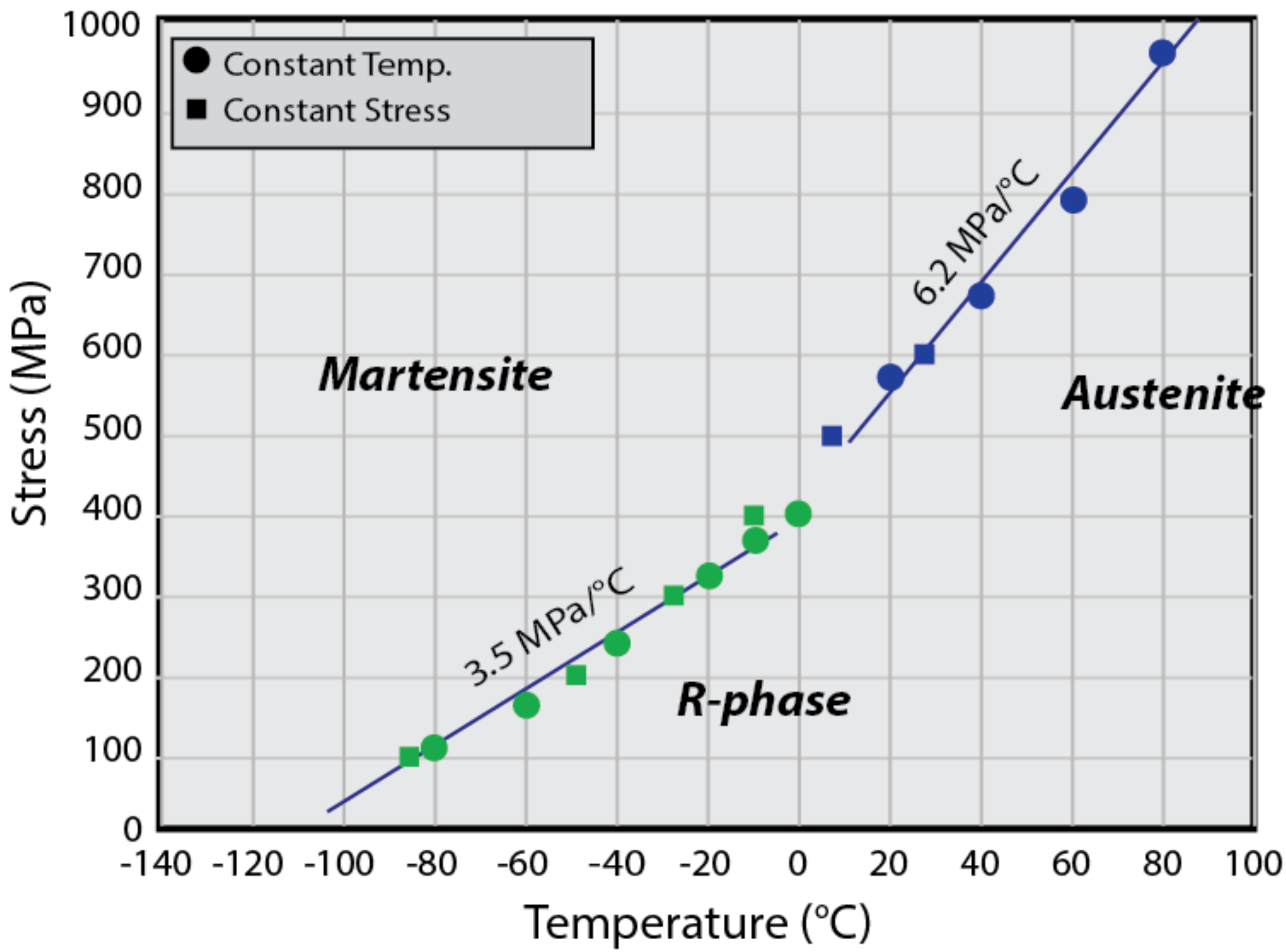


Determining whether the parent phase is Austenite or R

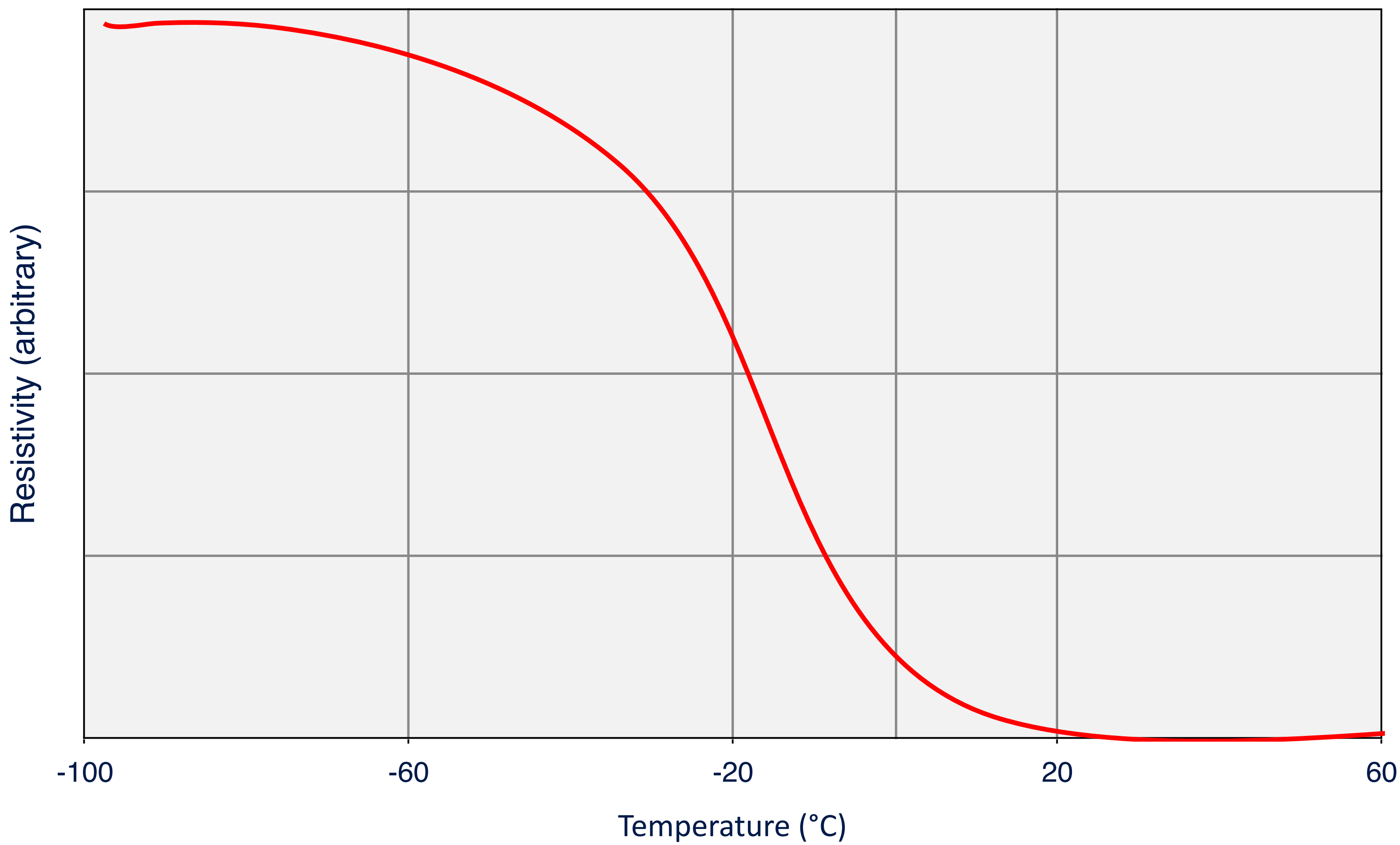




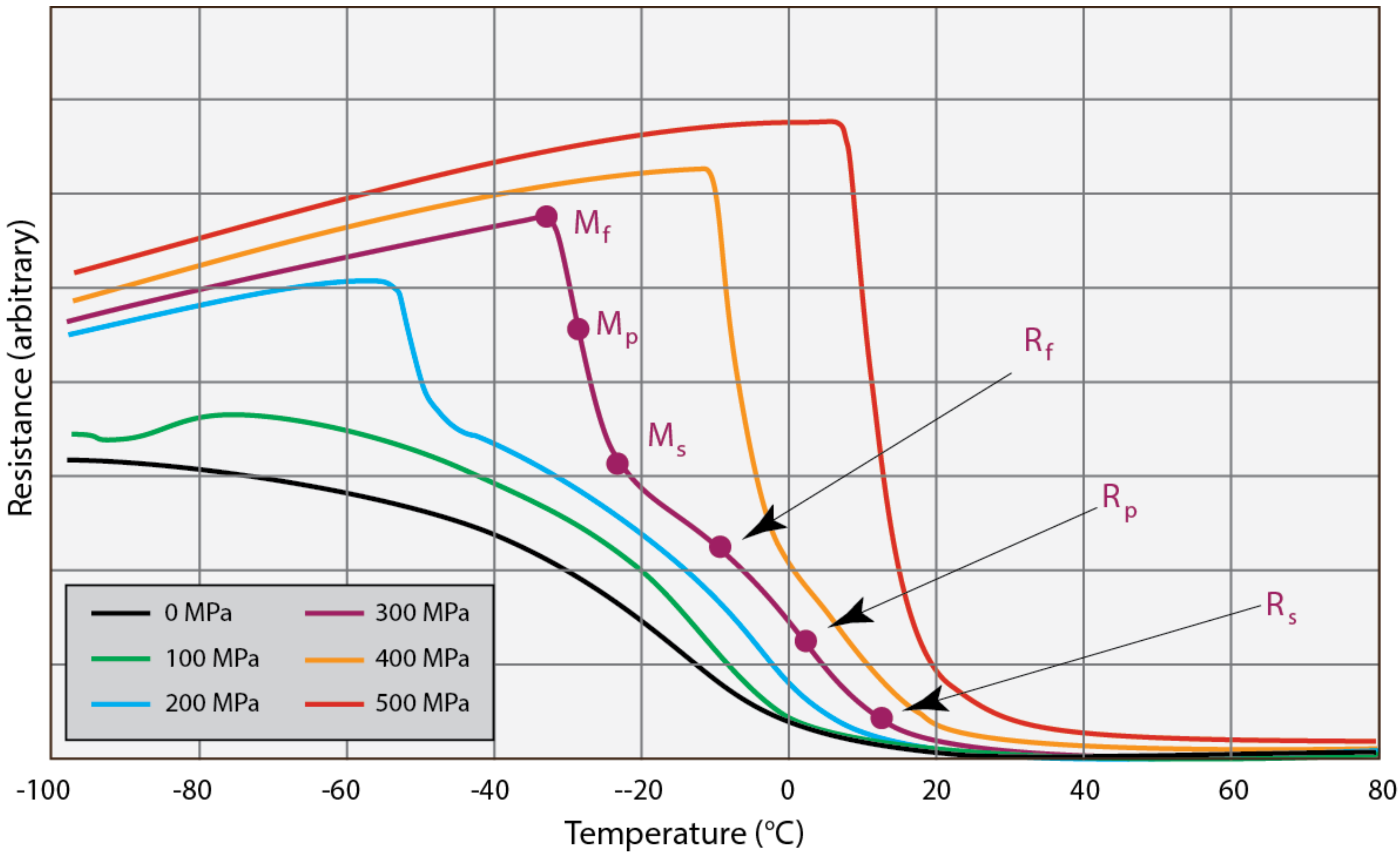
Forward transformation to Martensite



Resistivity increases as R is formed from A

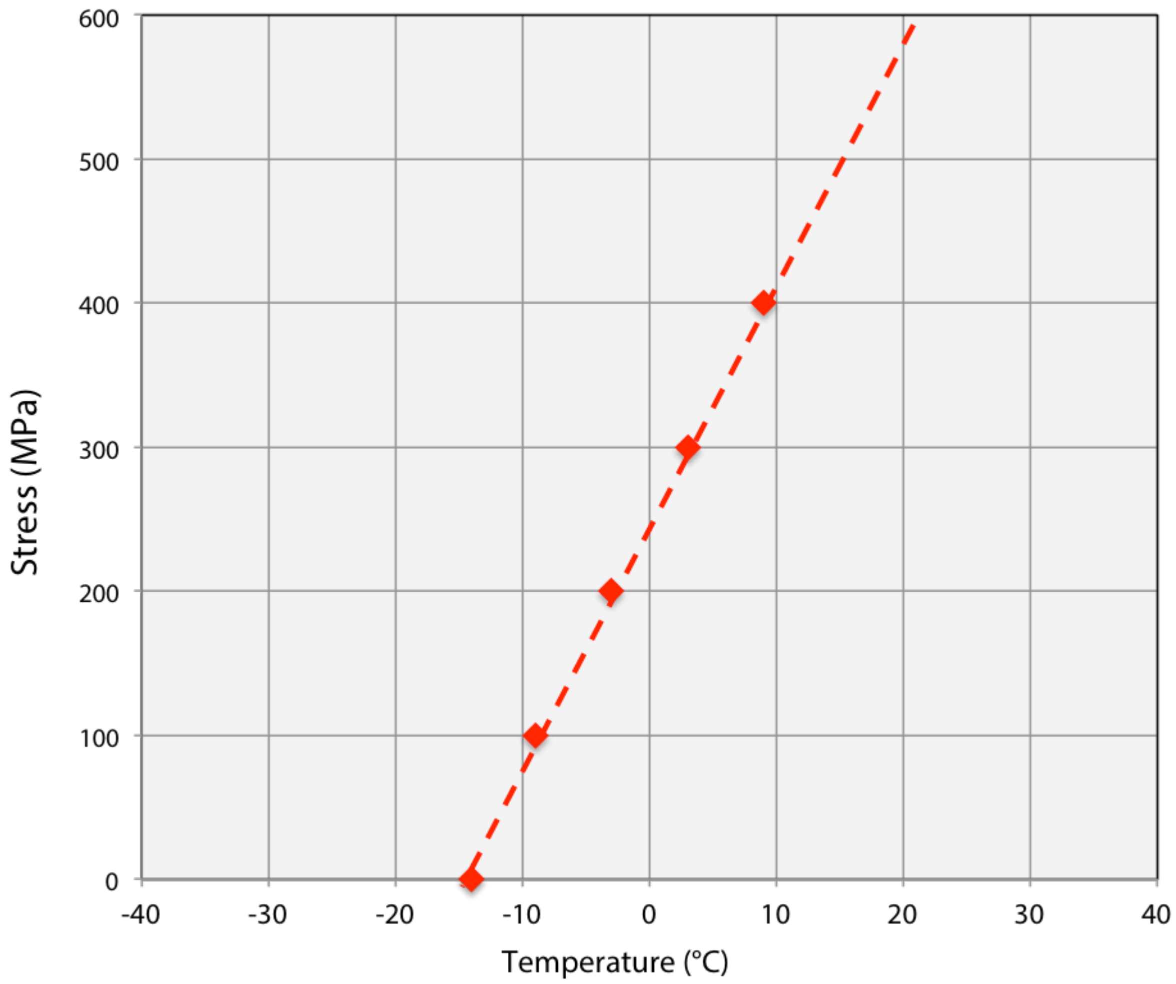


Resistance under constant stress maps the A-R transus*



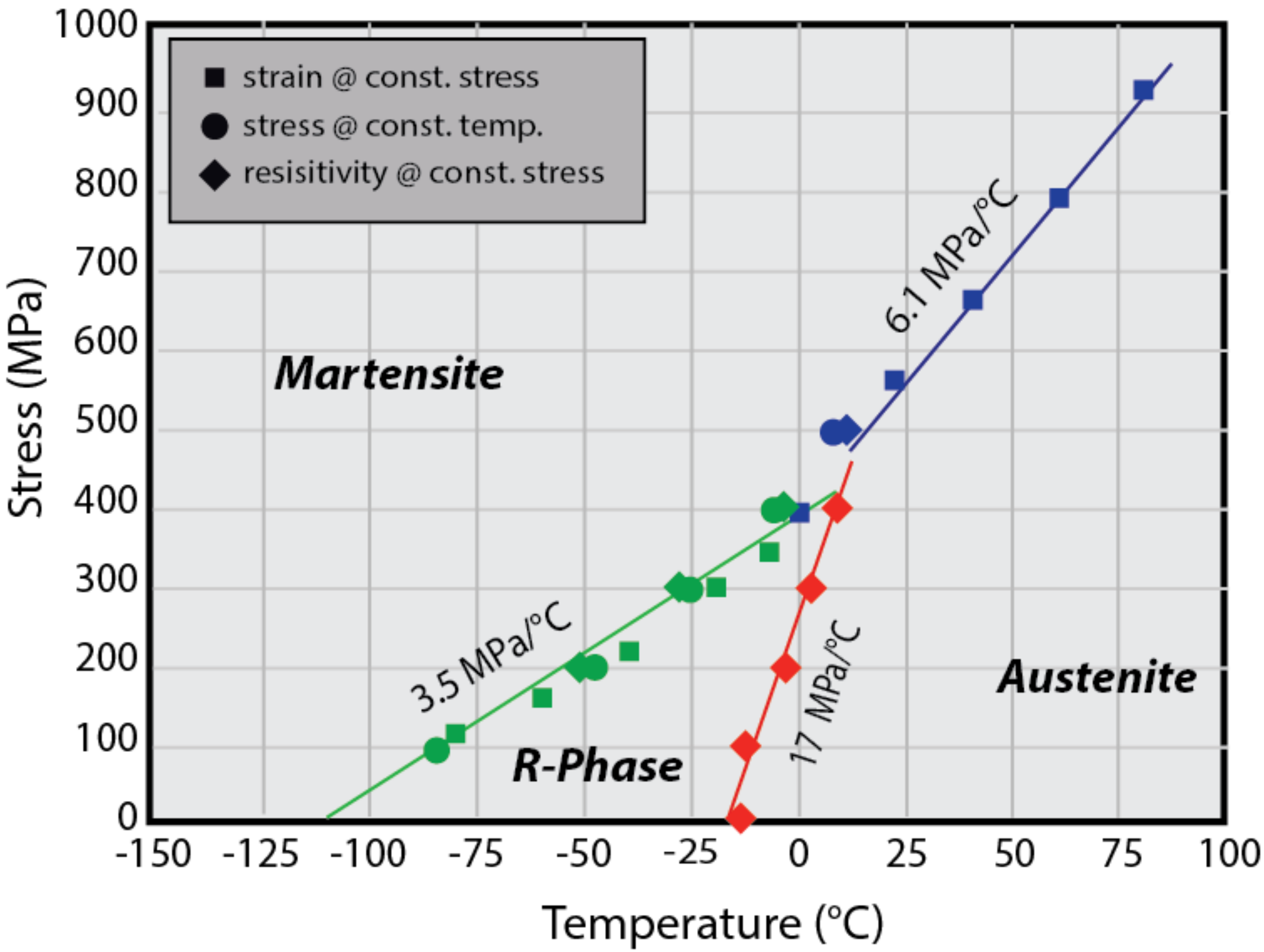
* Four point resistance method run in an load-controlled, screw-based testing machine

Forward transformation from A to R

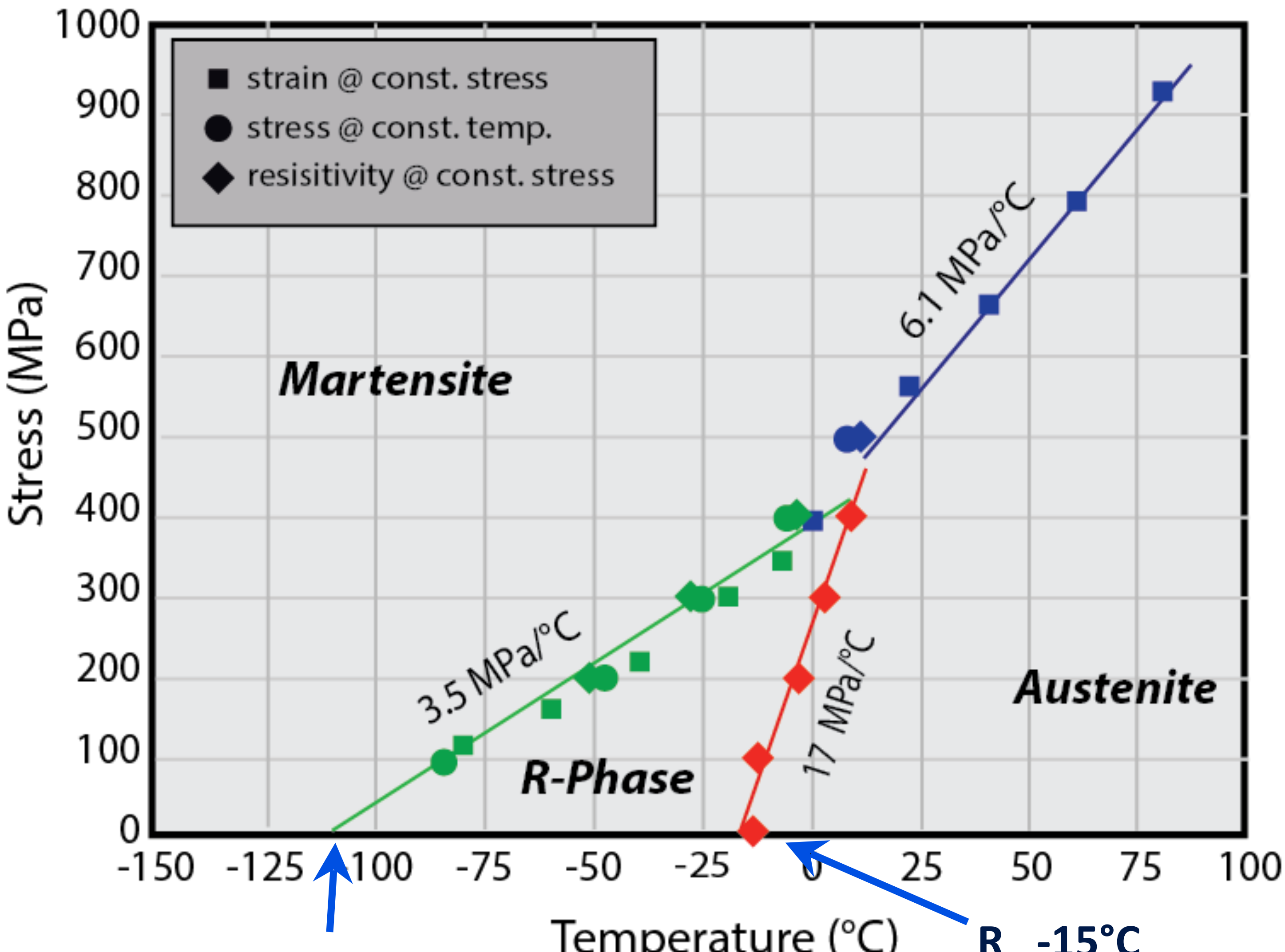


CONFLUENT

The full A-R-M phase diagram in the forward direction



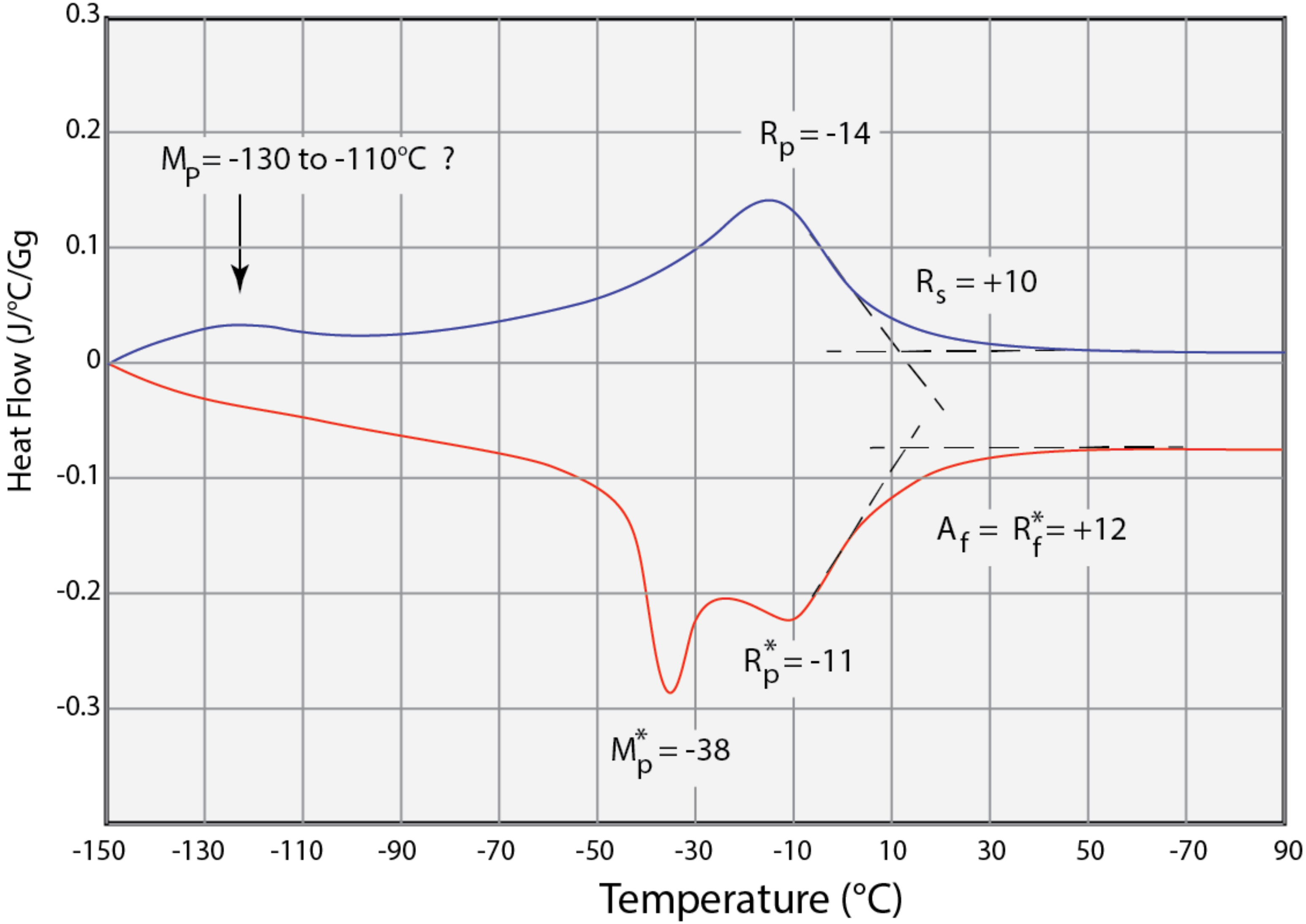
If the slopes are "crystallographically fixed," extrapolating to the zero-stress transformation temperatures defines the entire diagram



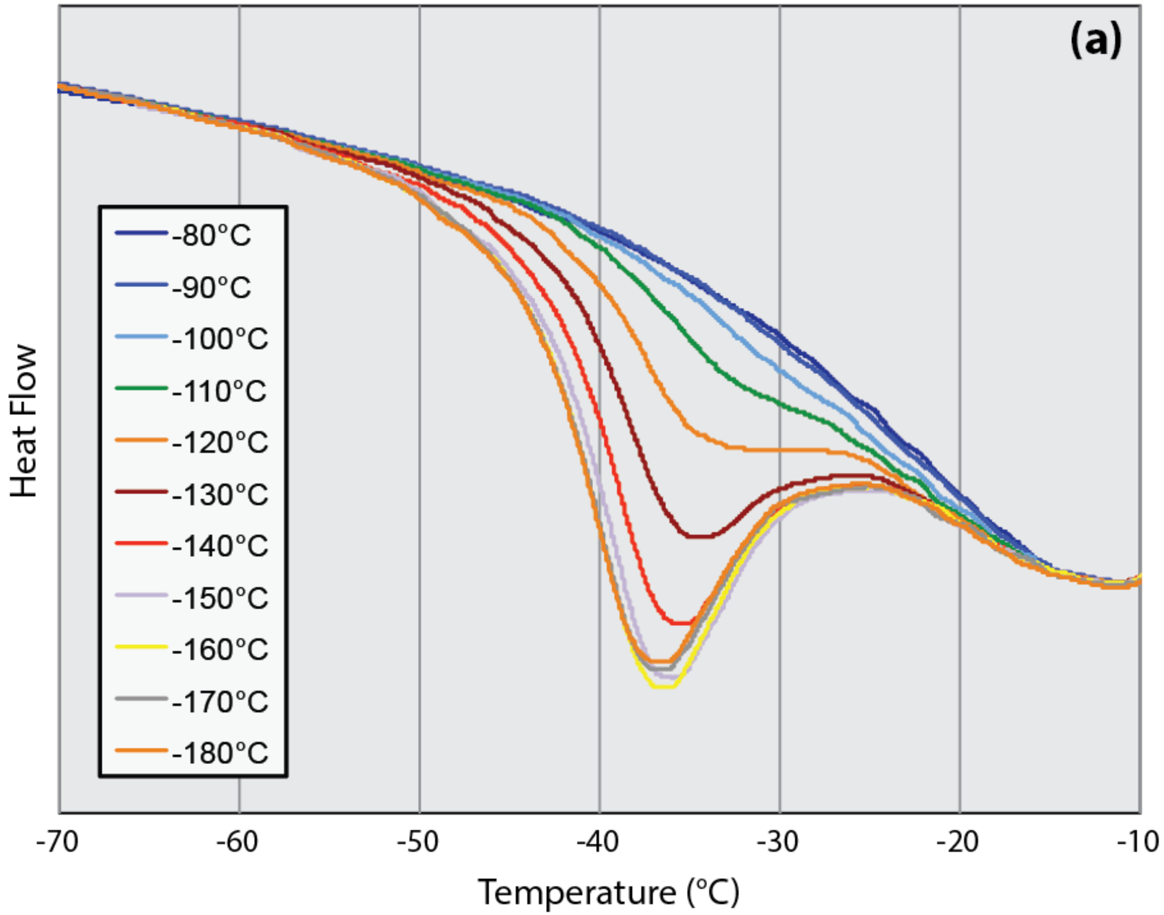
$M_{s,p}$=-110°C

$R_{p=}$-15°C

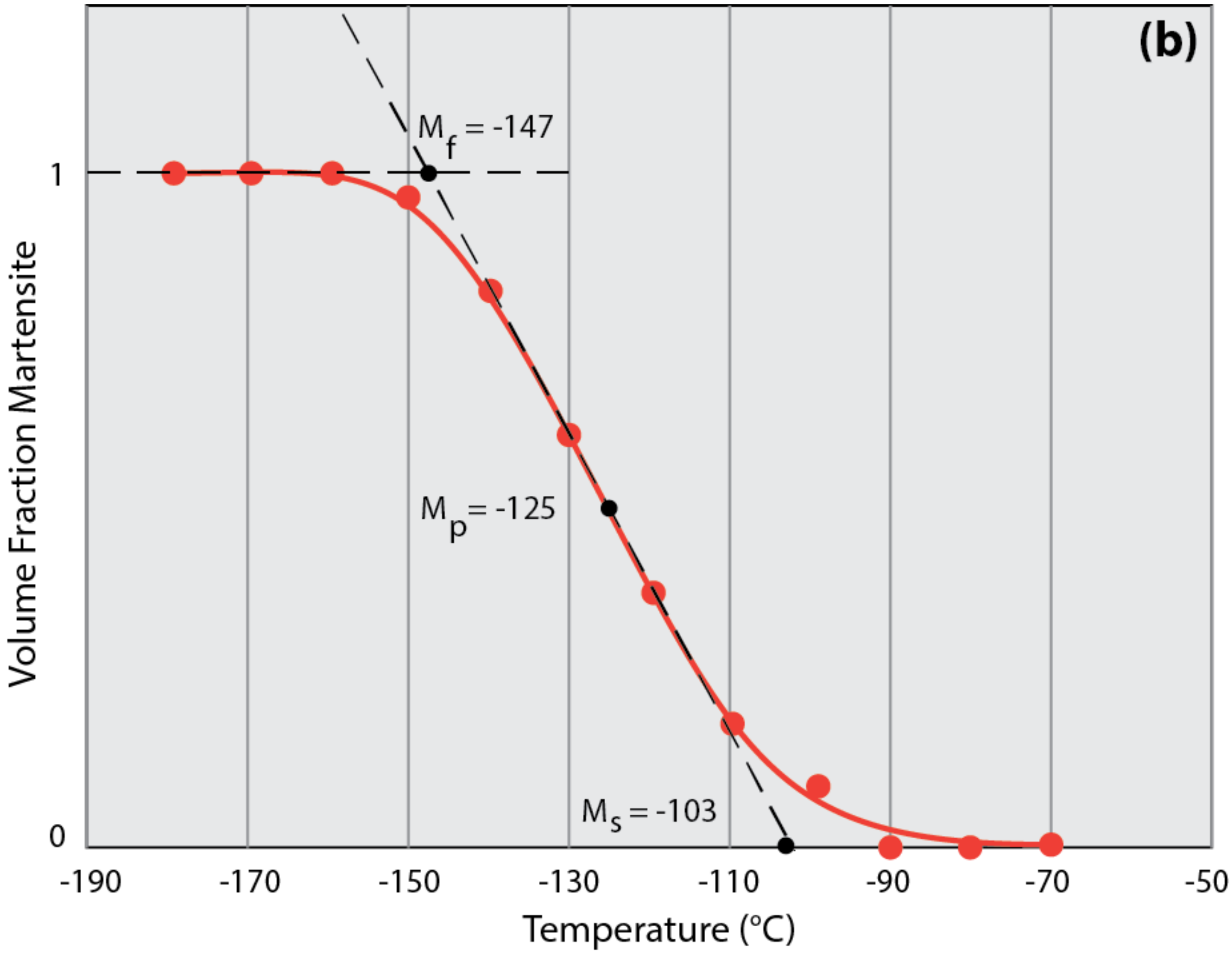
DSC: (forward transformation in blue)



Tracking martensite reversion signal after cooling to various temperatures

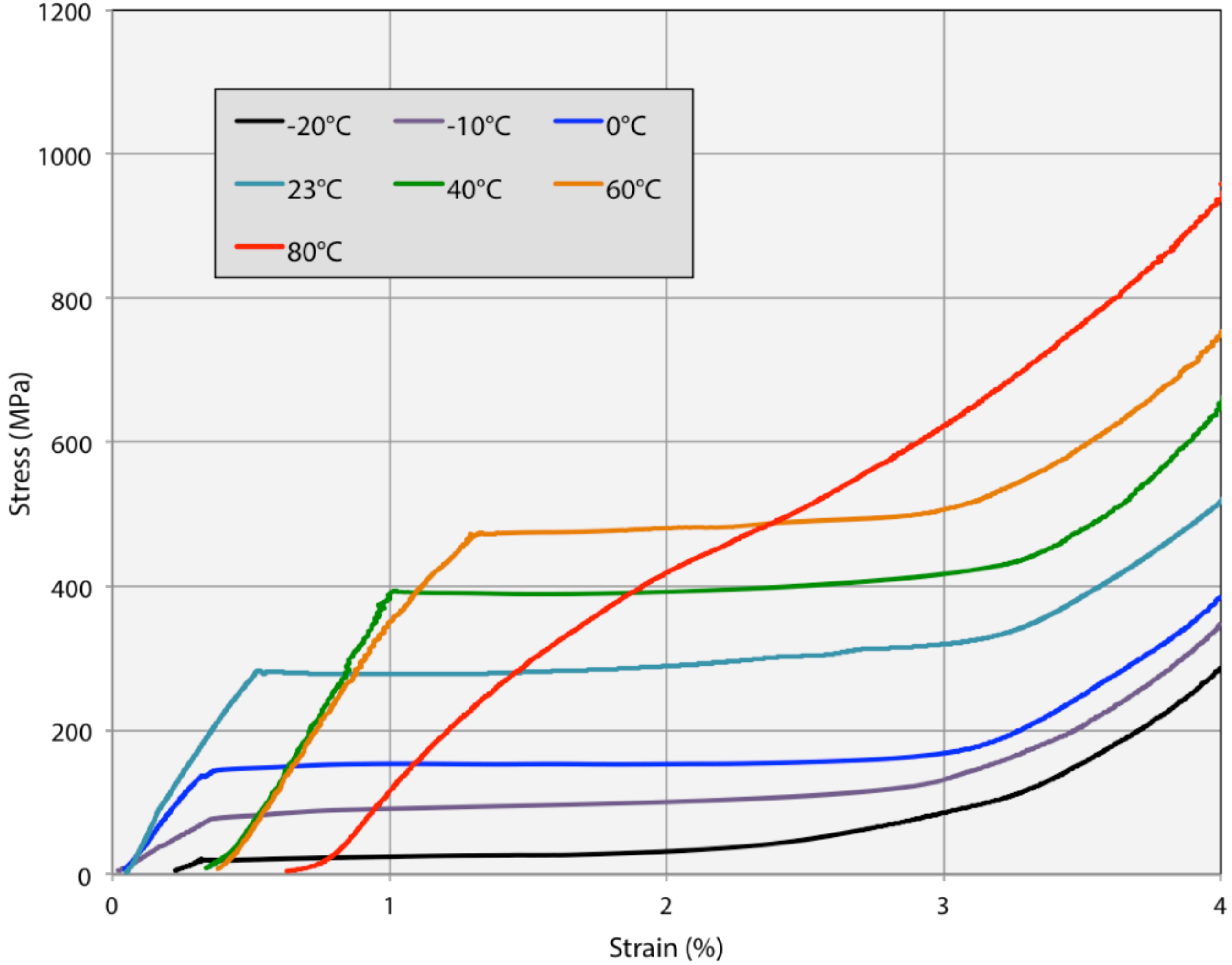


Integrating the reversion peaks allow one to identify martensite formation temperatures



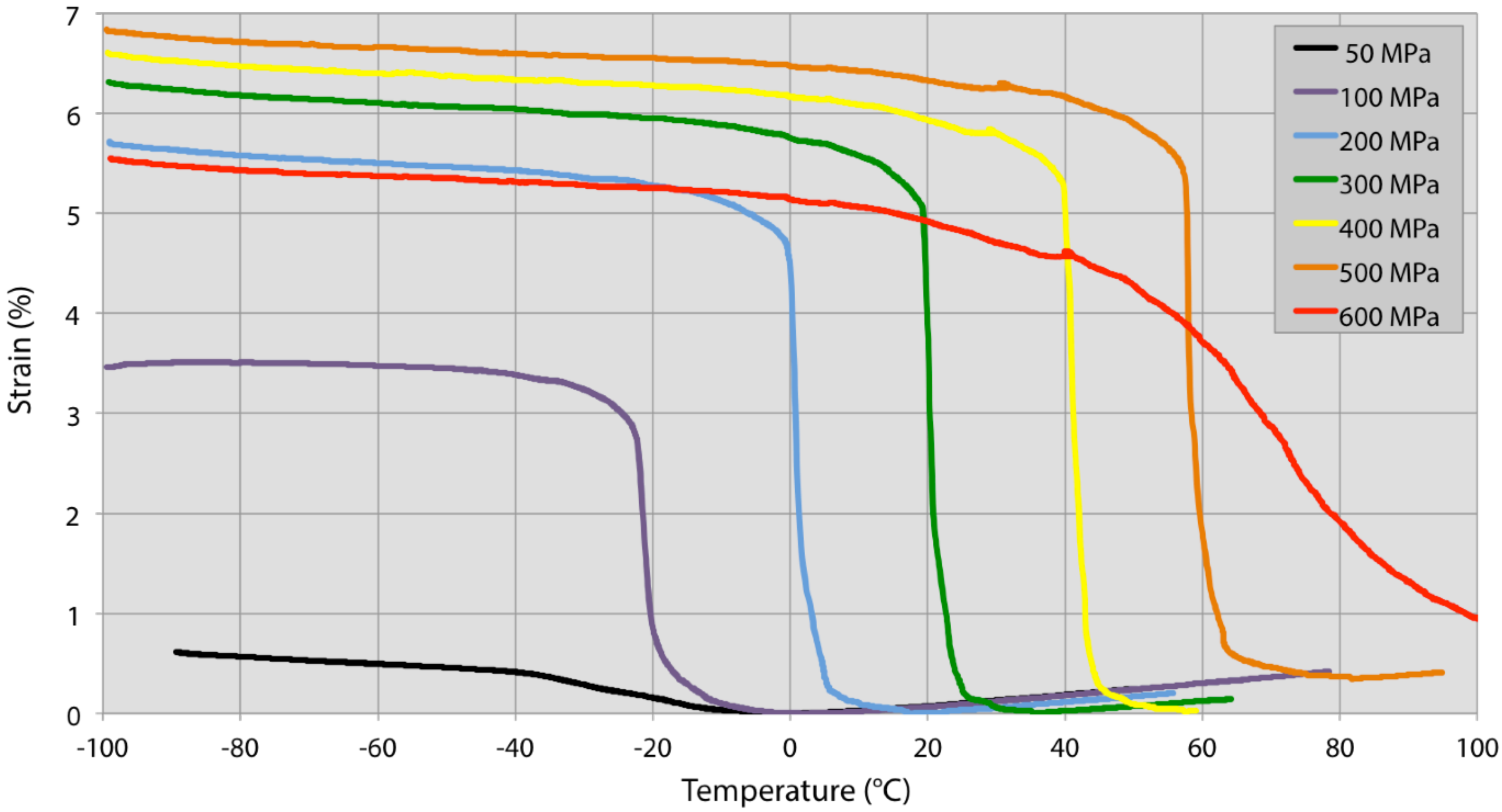
Moving on to the reverse transformation: (Toward increased entropy)

Unloading plateaus (Martensite reversion… not necessarily Austenite formation)

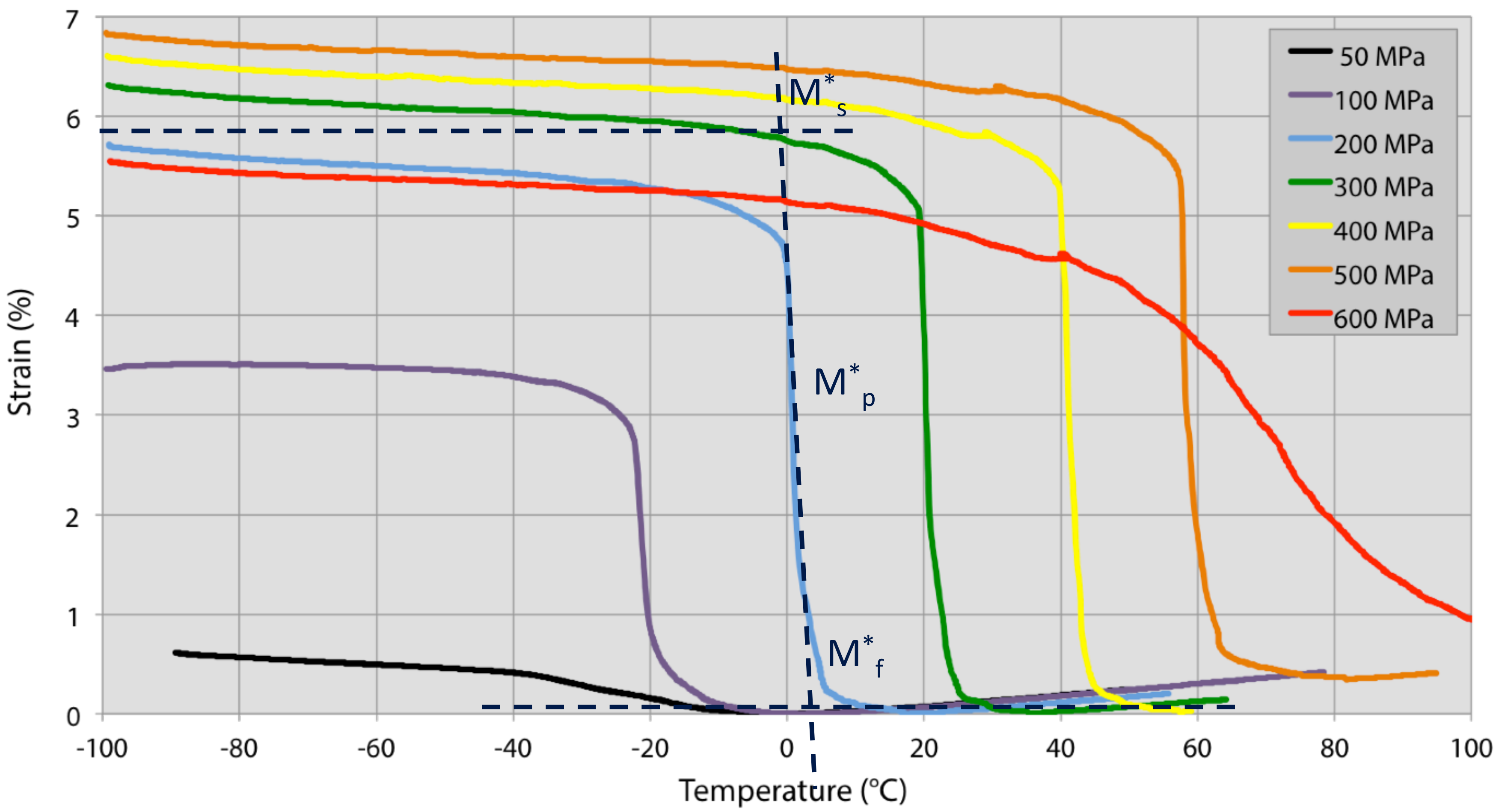


CONFLUENT

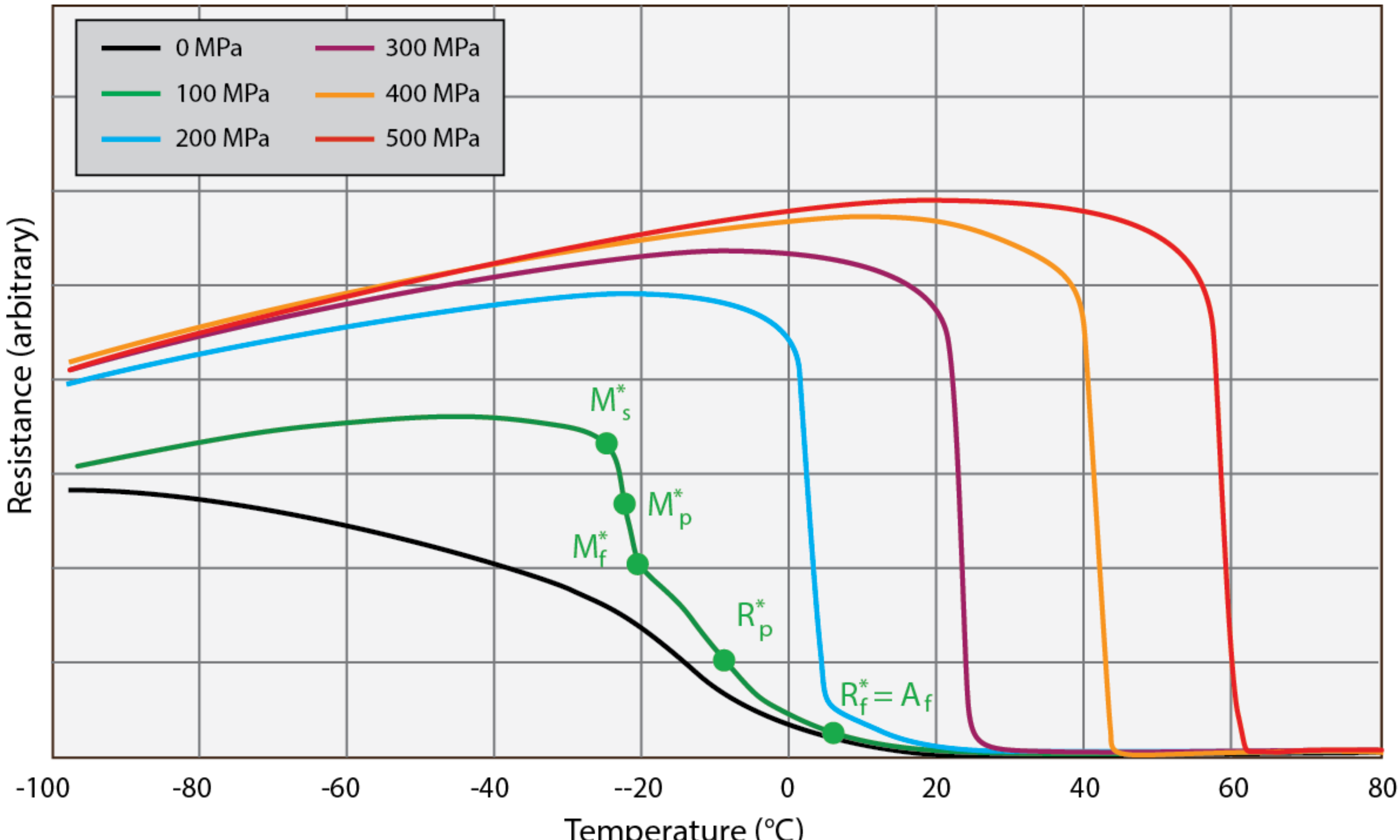
Heating under constant load



M* refers to the temperature at which Martensite reverts, regardless of whether it reverts to R or A



Using M* terminology rather than R'



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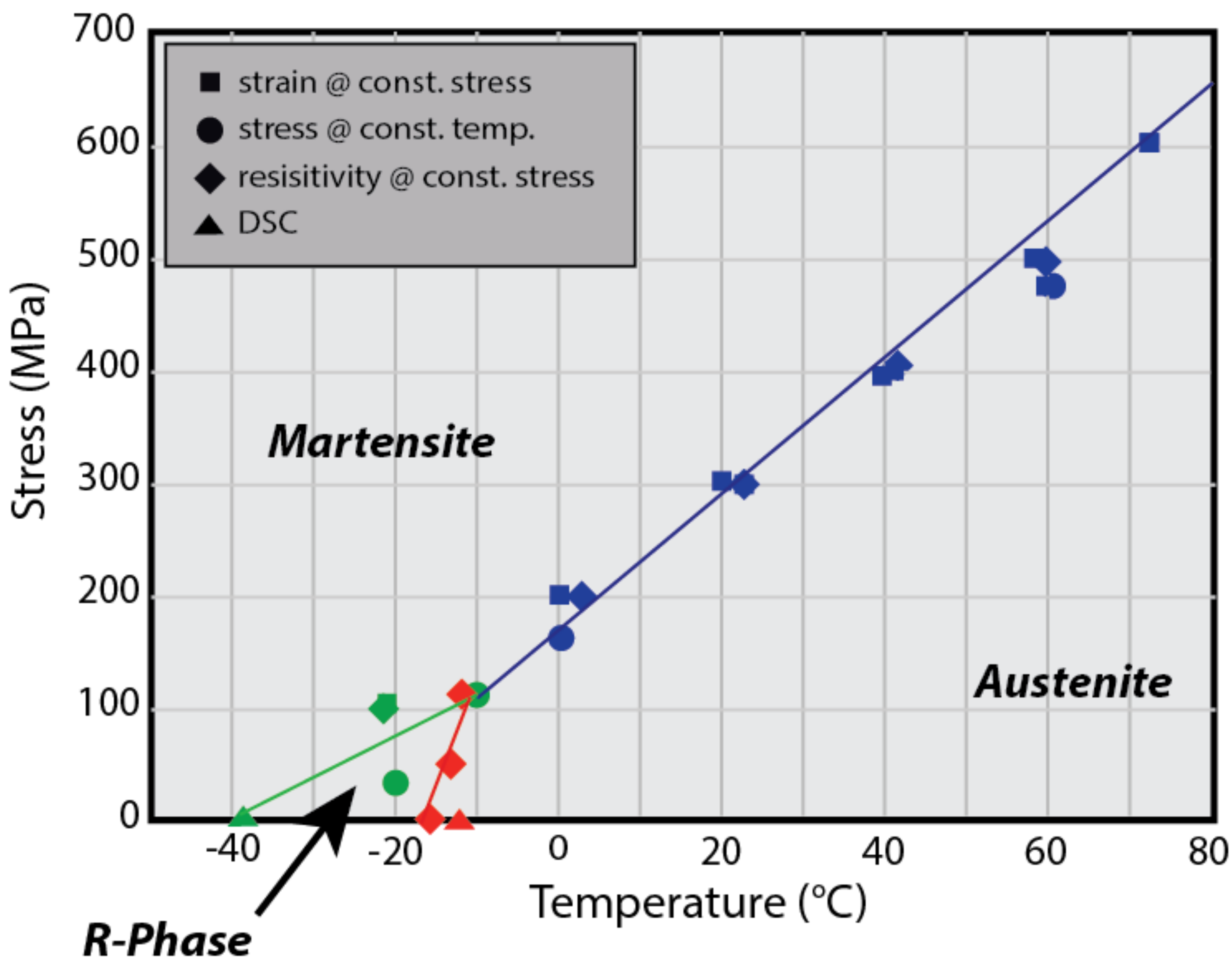
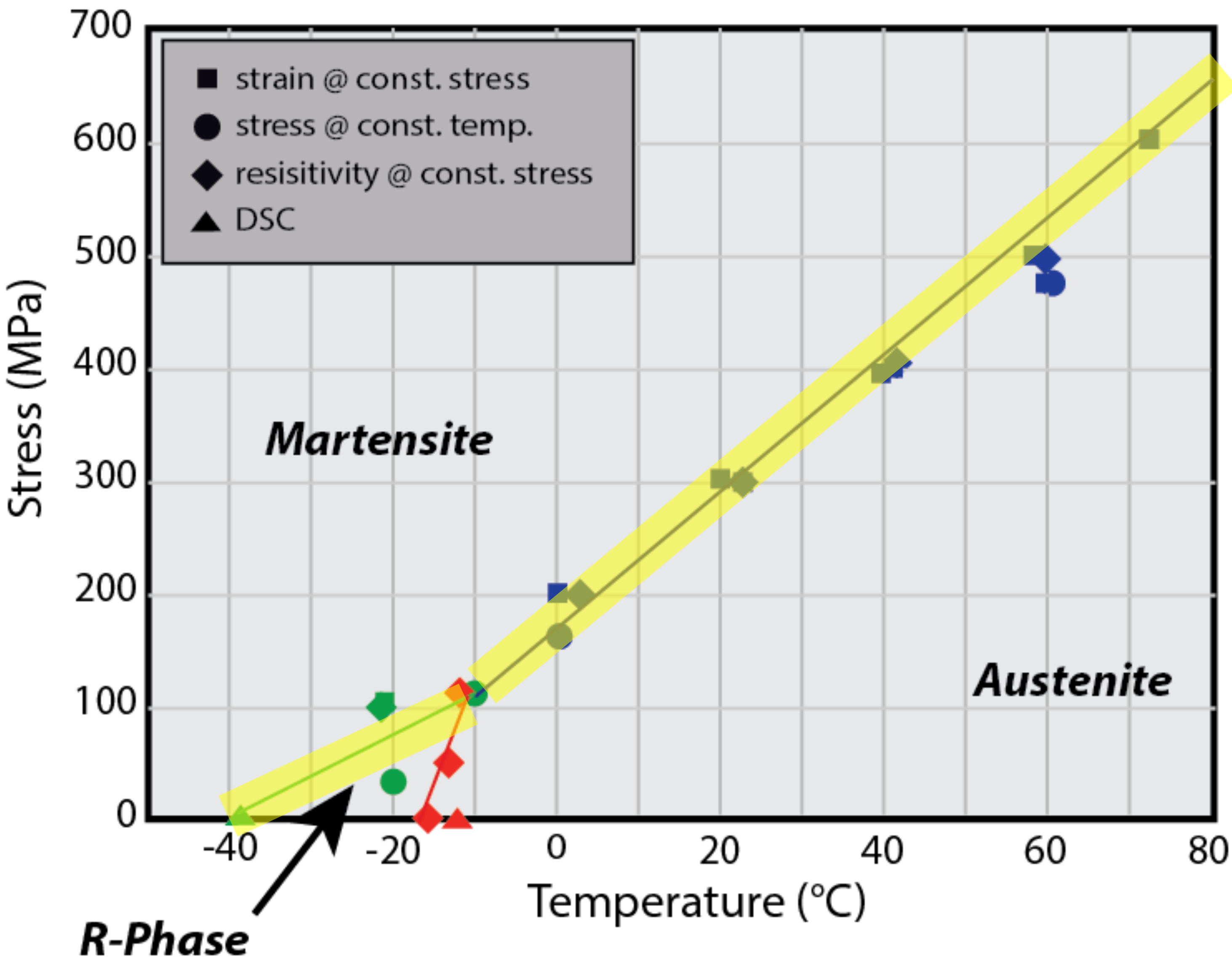
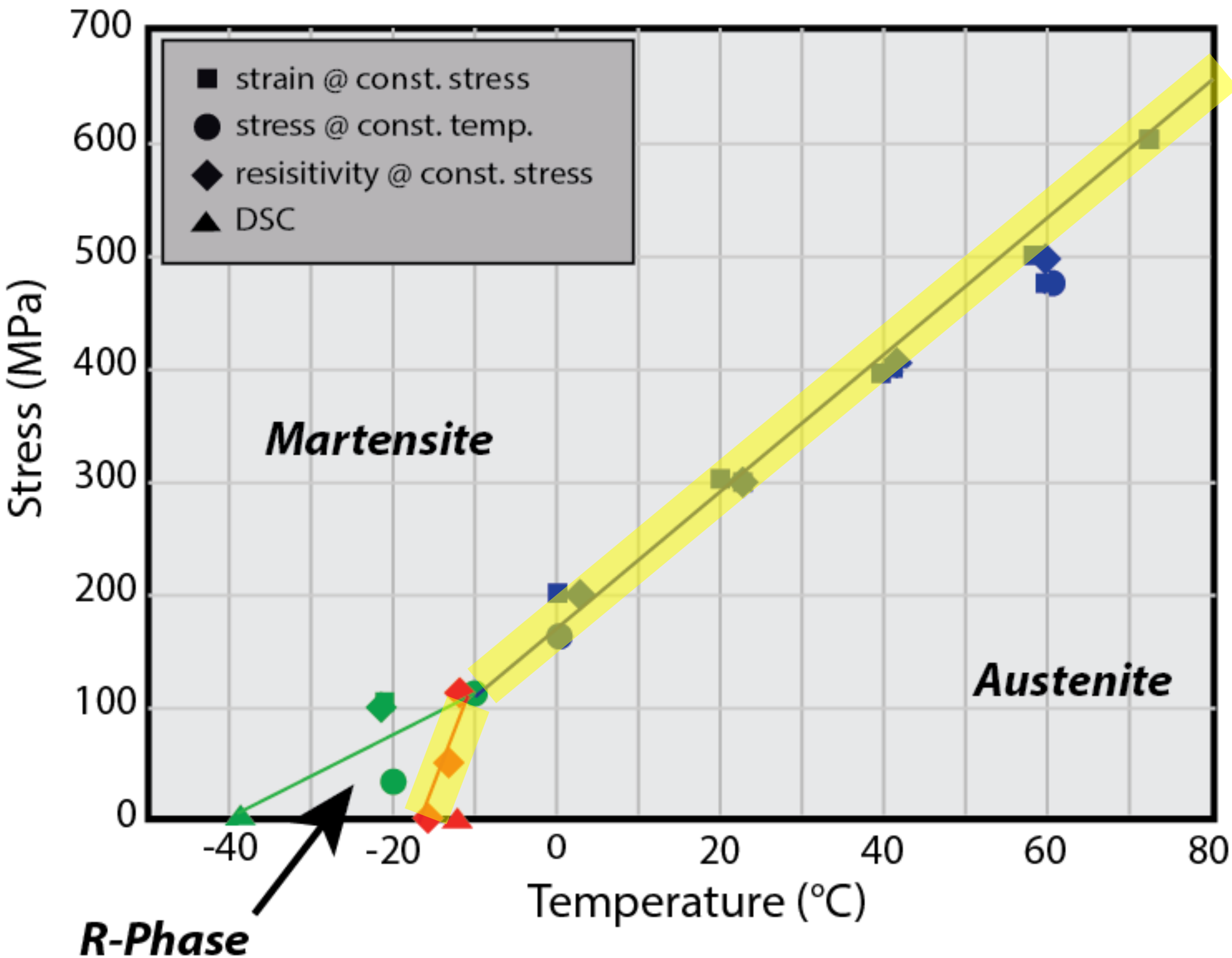
Reverse transformation phase diagram



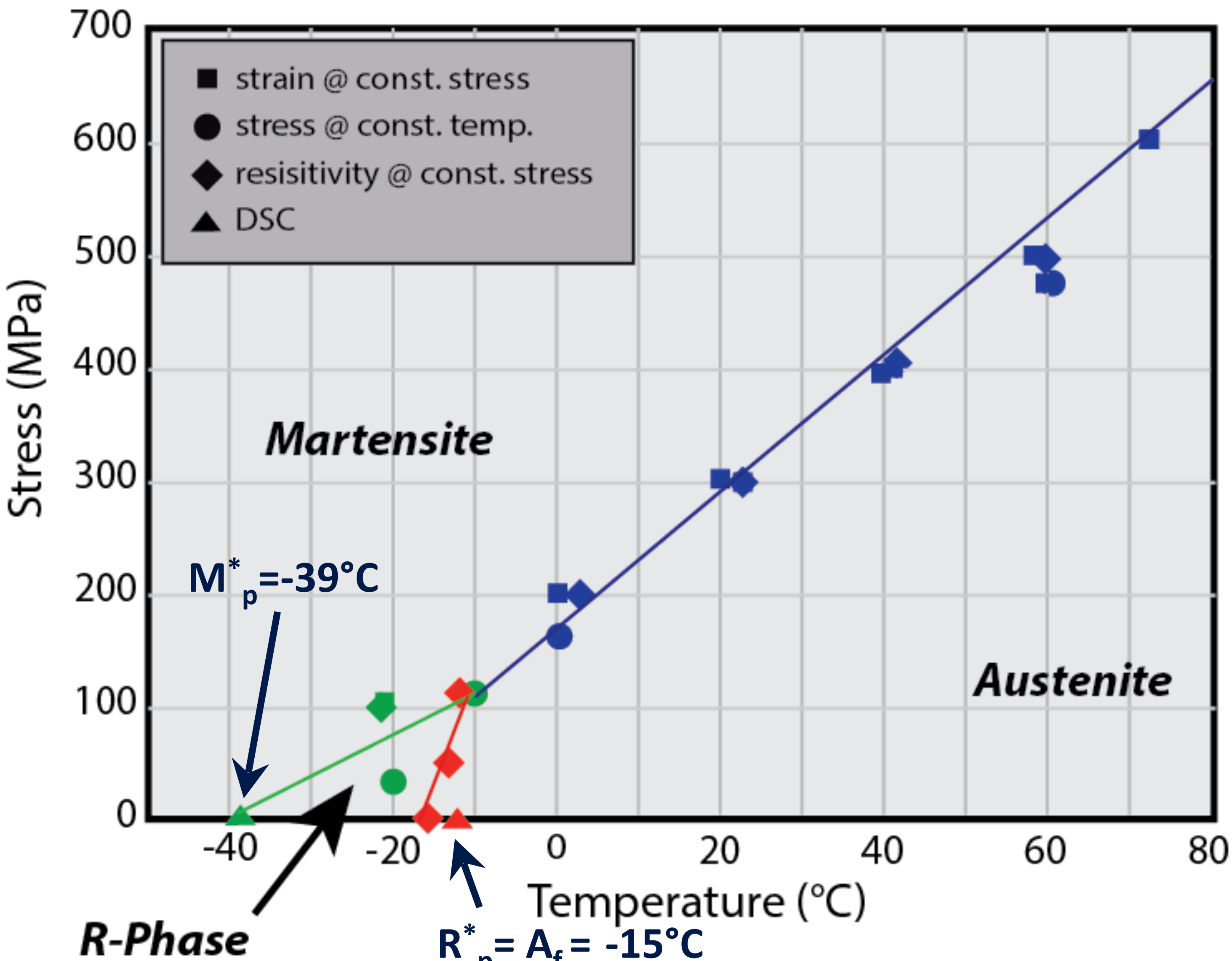
M* - Martensite reversion



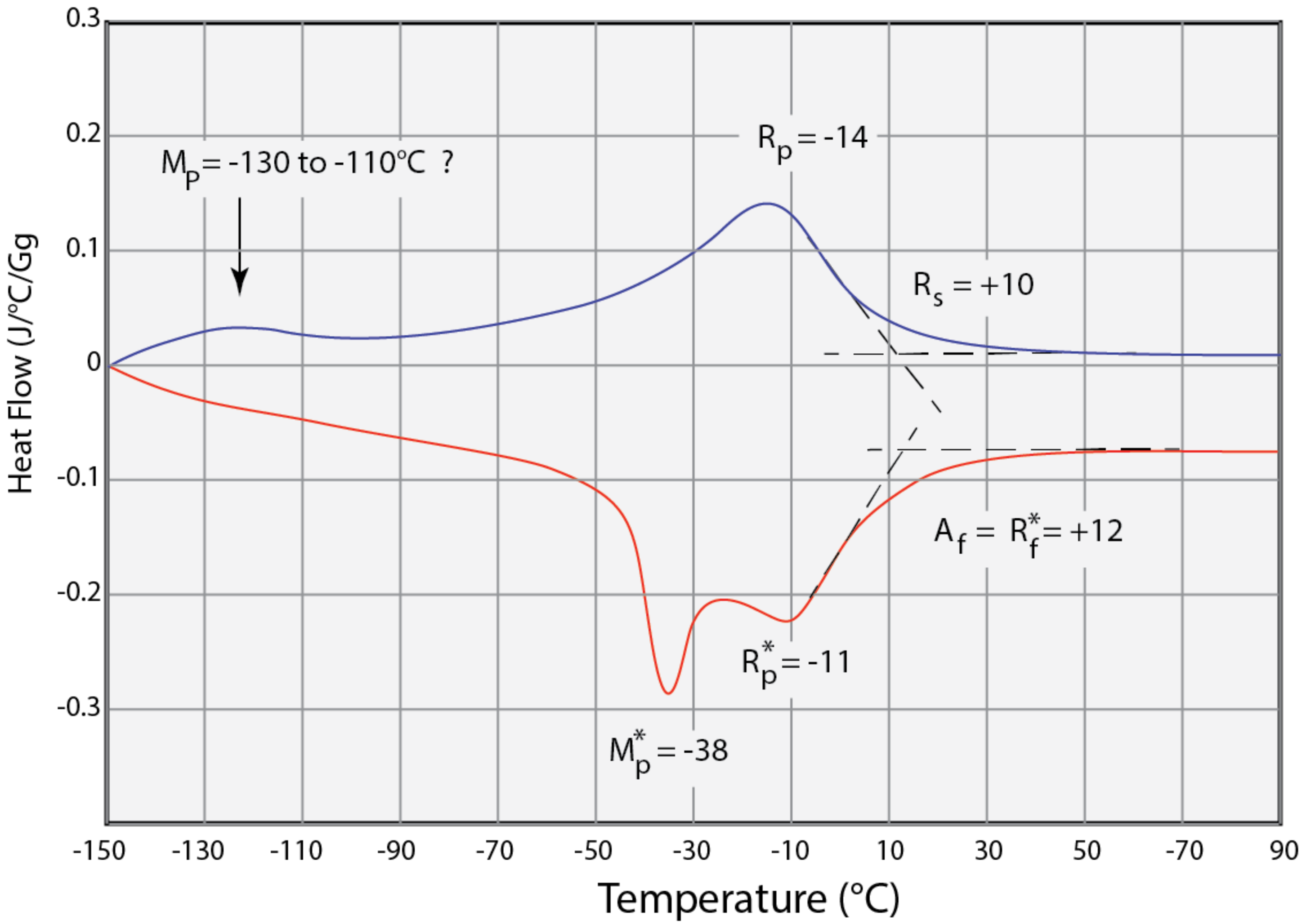
A_f - Austenite formation



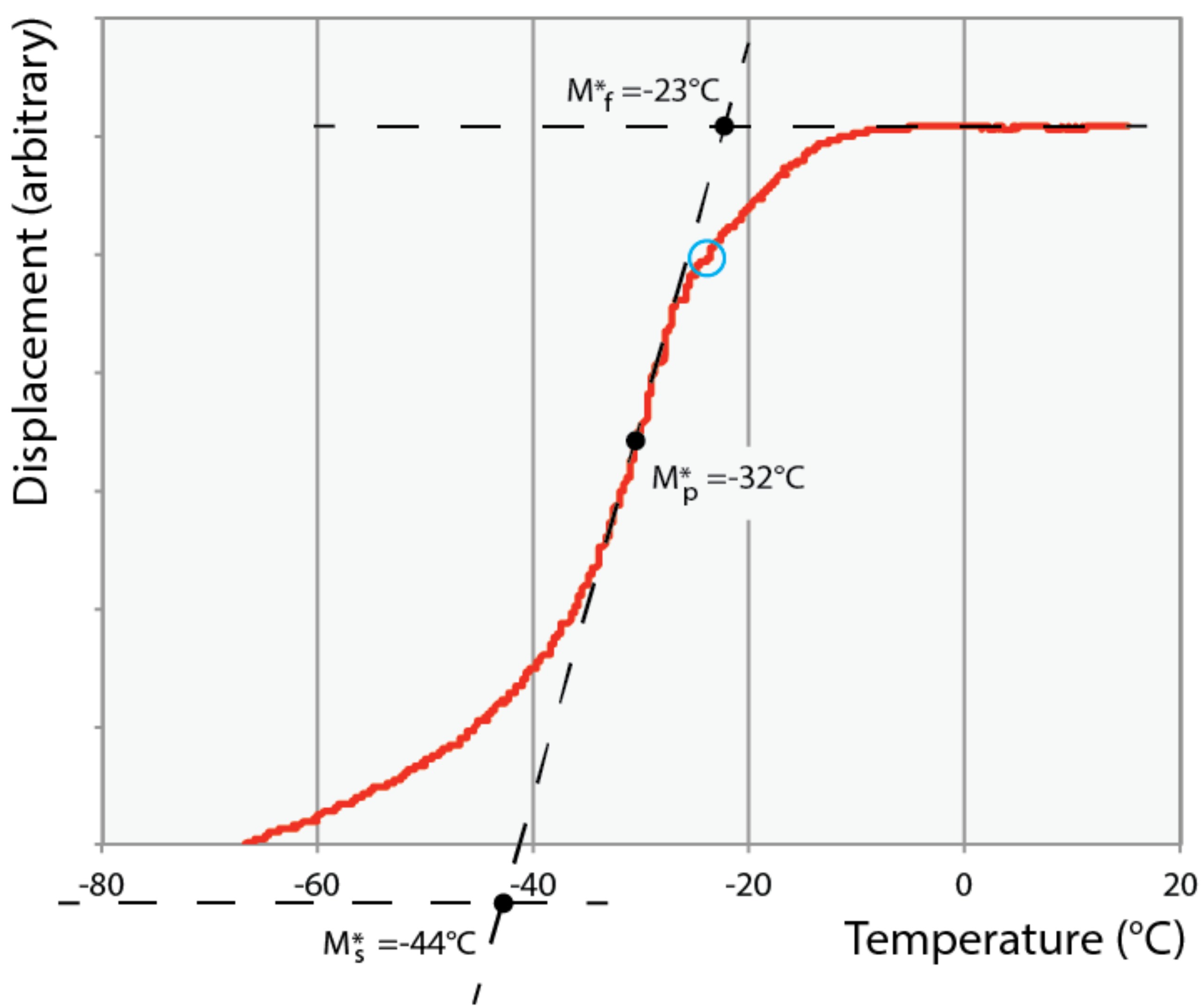
Extrapolated transformation temperatures



Are in perfect agreement with DSC

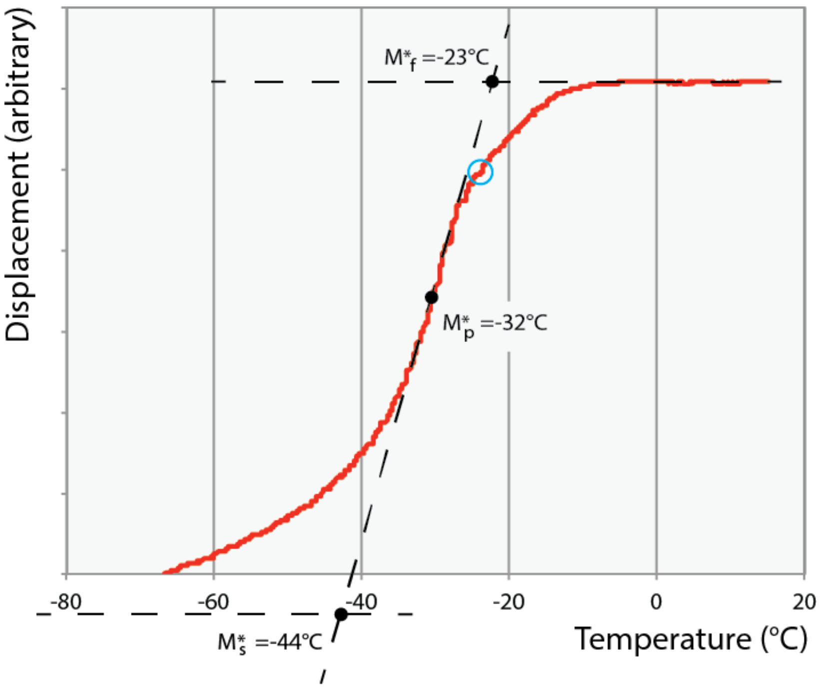


"Bend Free Recovery" agrees roughly, but not as well

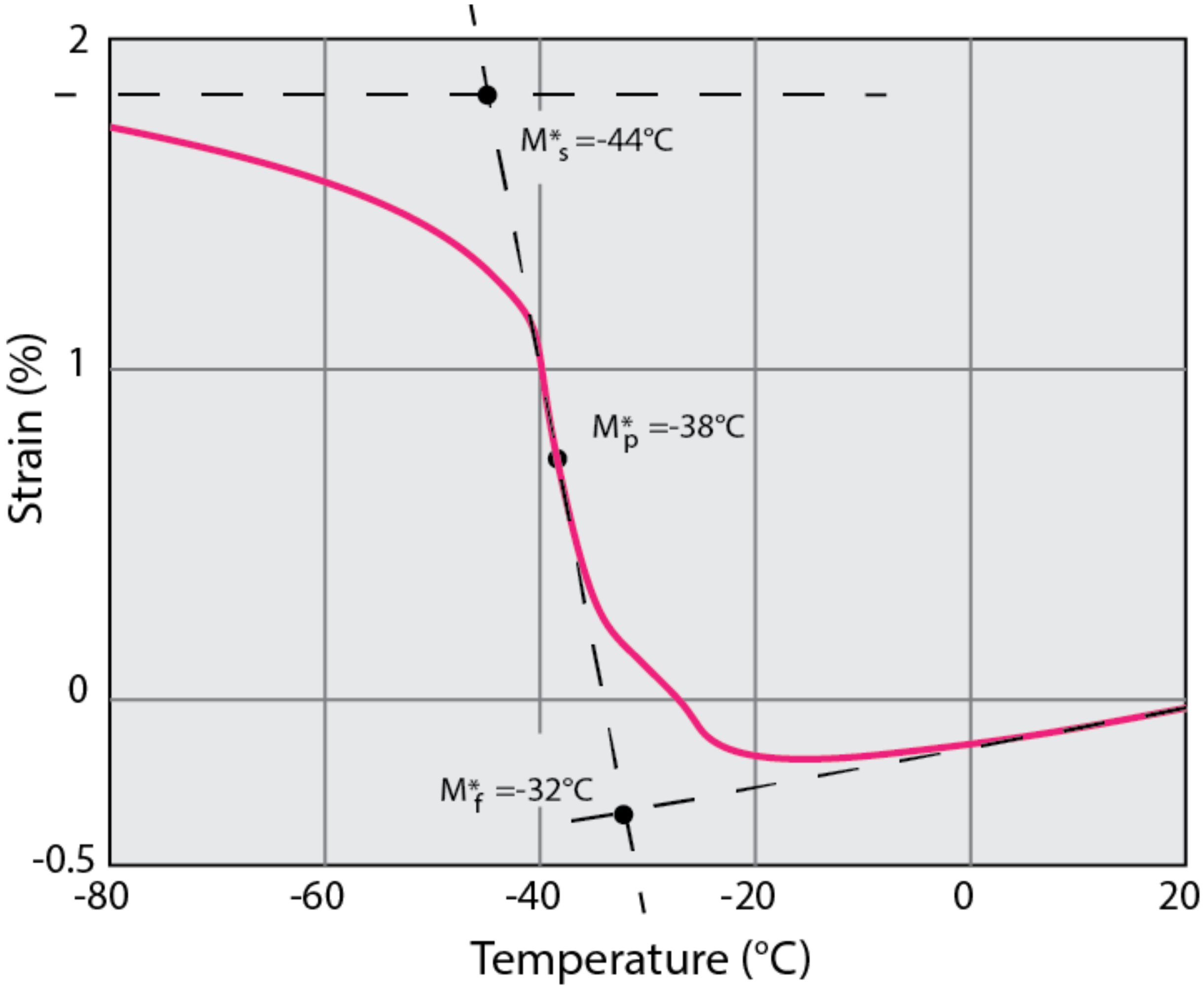


Limitations of "Bend Free Recovery"

- Compression/Tension creates inhomogeneous dσ/dT
- Non-uniform deformation creates residual stresses
- Two percent strain at the outer fiber might mean only less than 5% volume fraction overall
- No strain localization

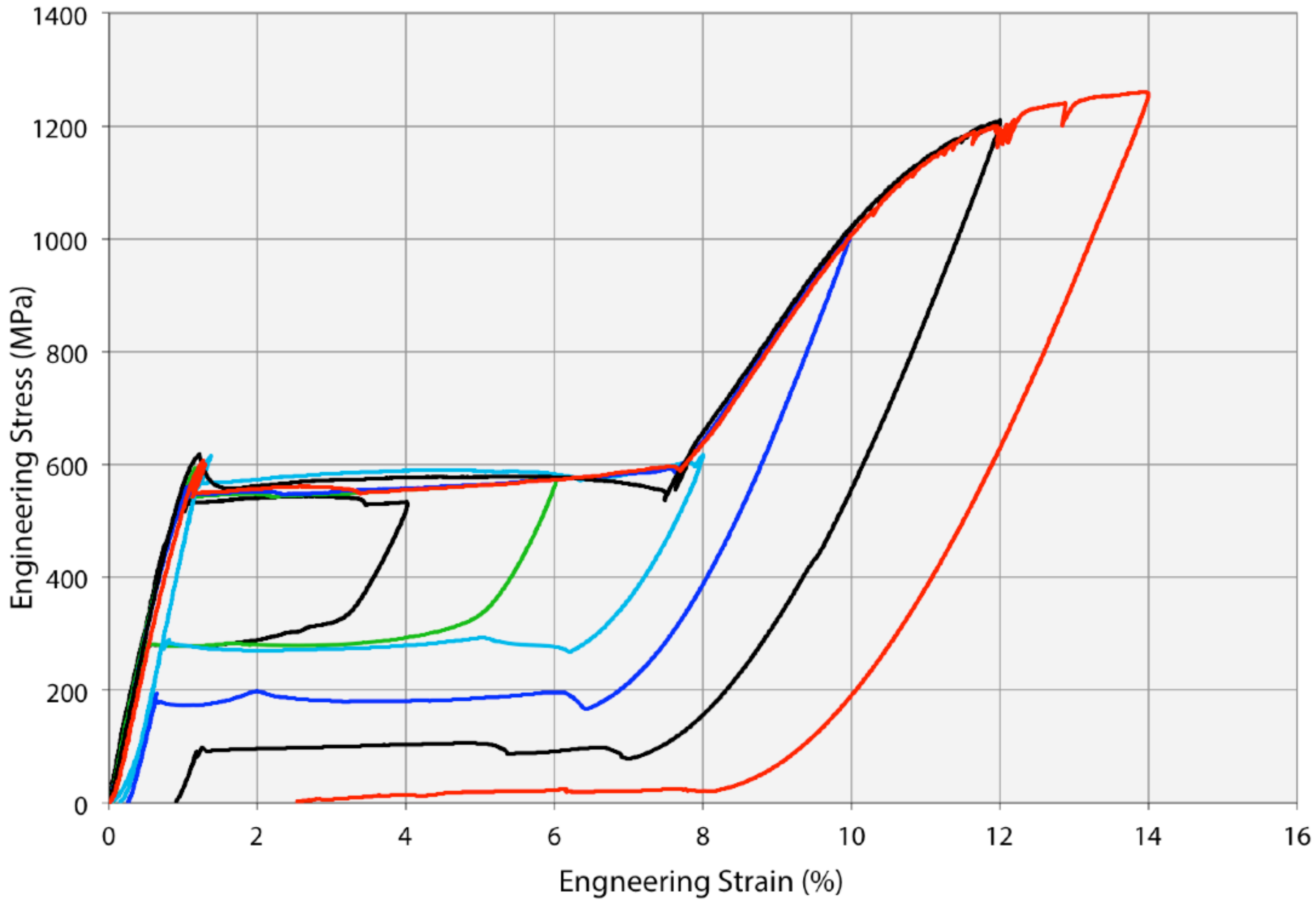


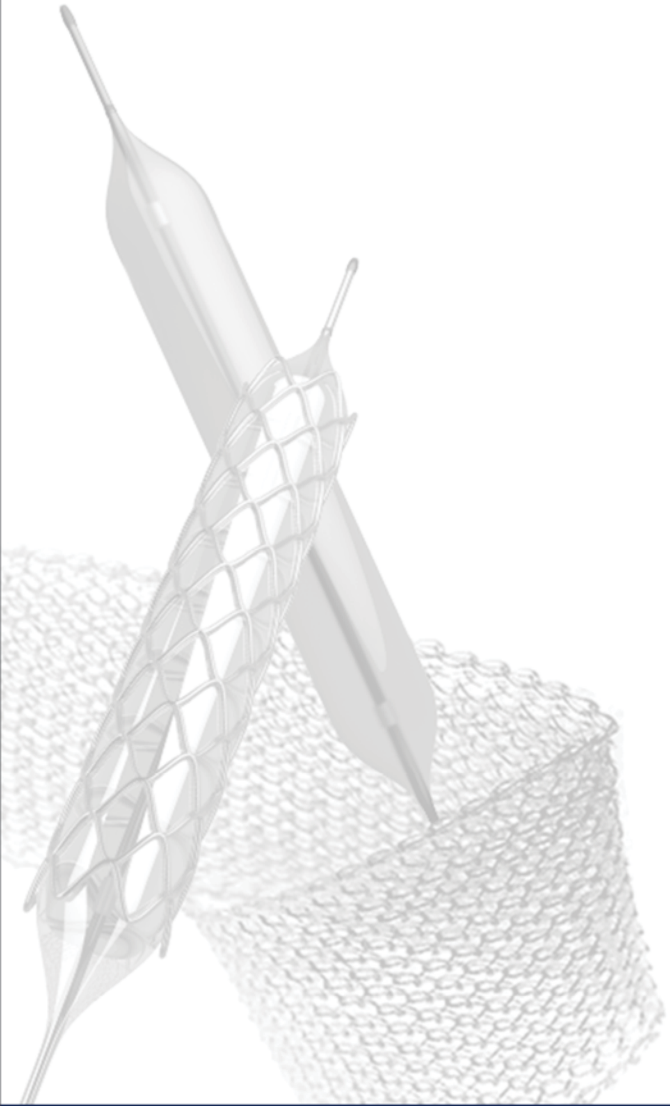
Tensile free recovery test resolves most of these issues*



* Deformed 2% in tension at -100°C, released, and warmed at 5°C/min.
Strain monitored by video extensometer

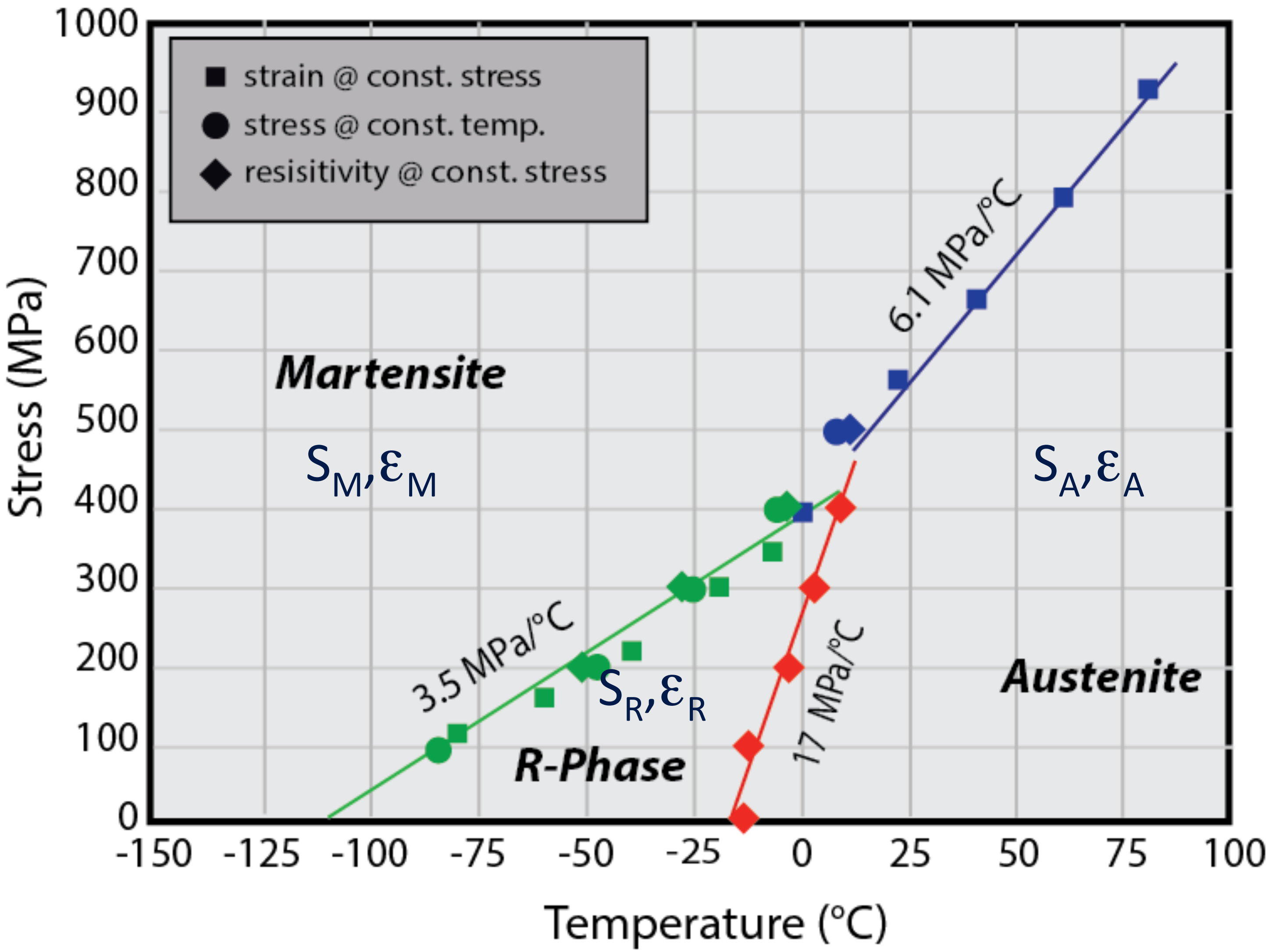
Caution:
Reverse transformation phase diagram is strain dependent





$d\sigma/dT$

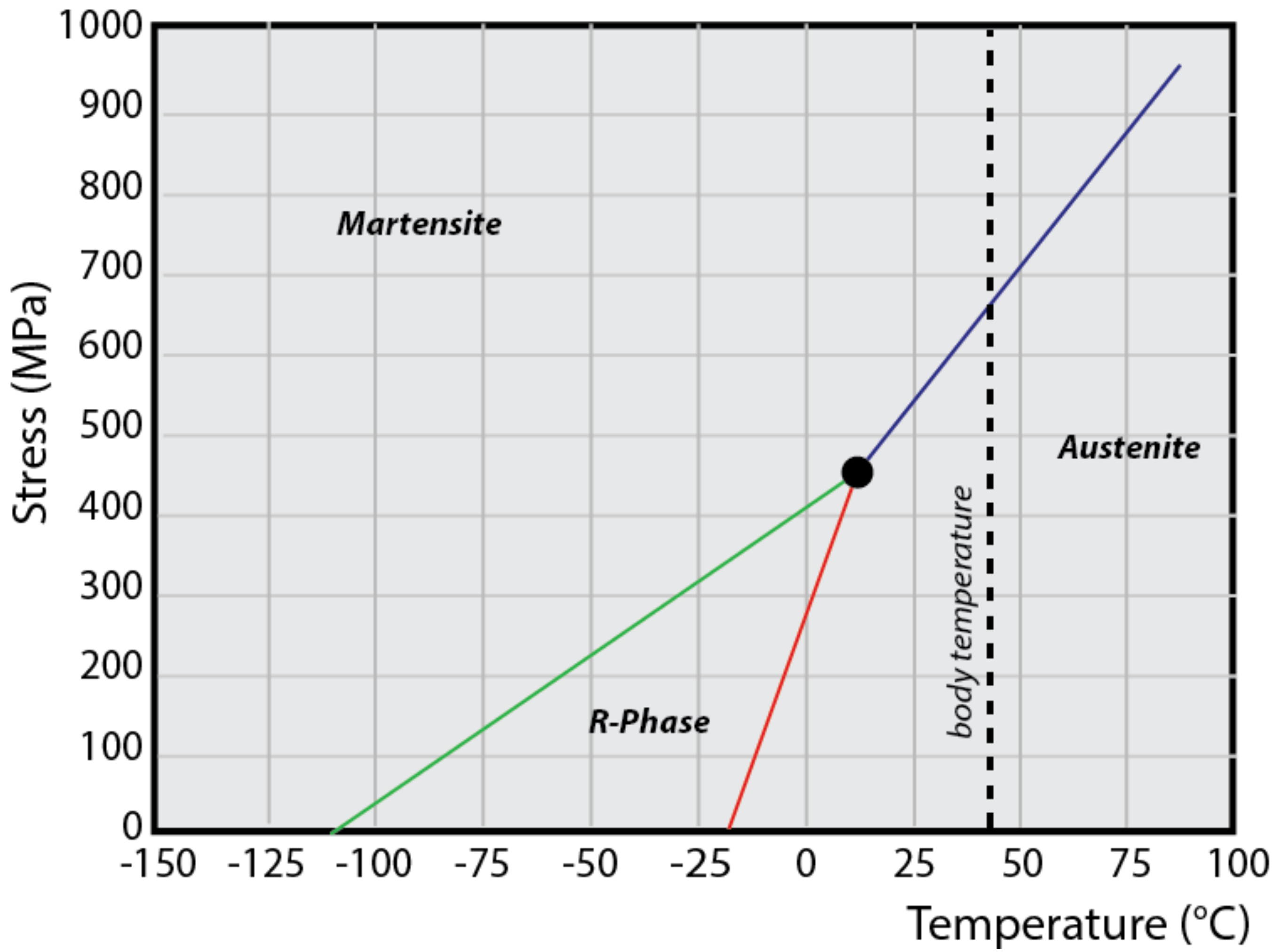
Phase diagram slopes are defined by the entropy and ability of each phase to change shape



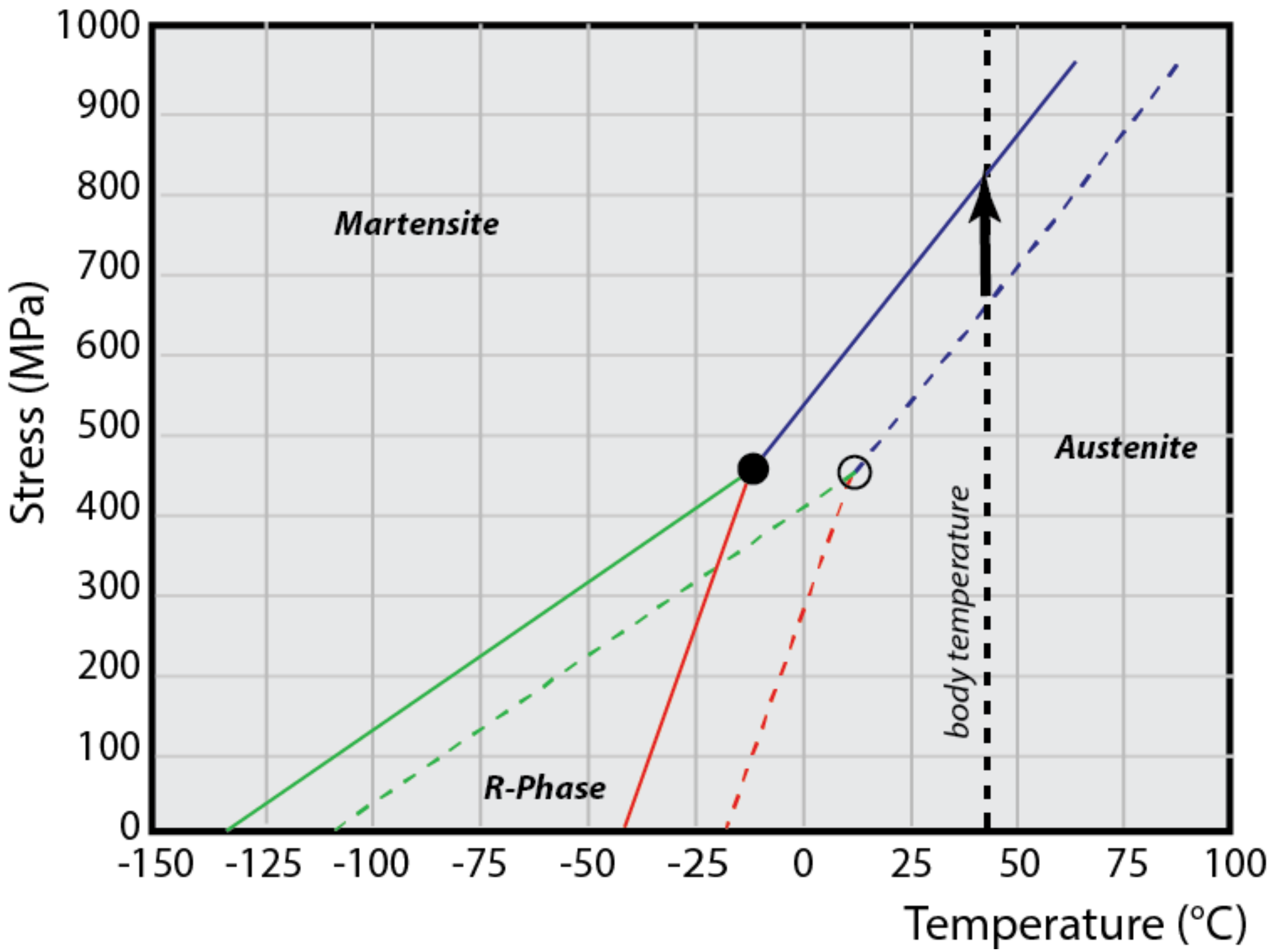
Each transus controlled by $(d\sigma/dT) = \Delta S / \Delta\varepsilon$, but affected by:

- Loading mode affects $\Delta\varepsilon$
- Texture affects $\Delta\varepsilon$
- Ni content affects $\Delta\varepsilon$ and ΔS
- Relative moduli of two phases affects $\Delta\varepsilon$

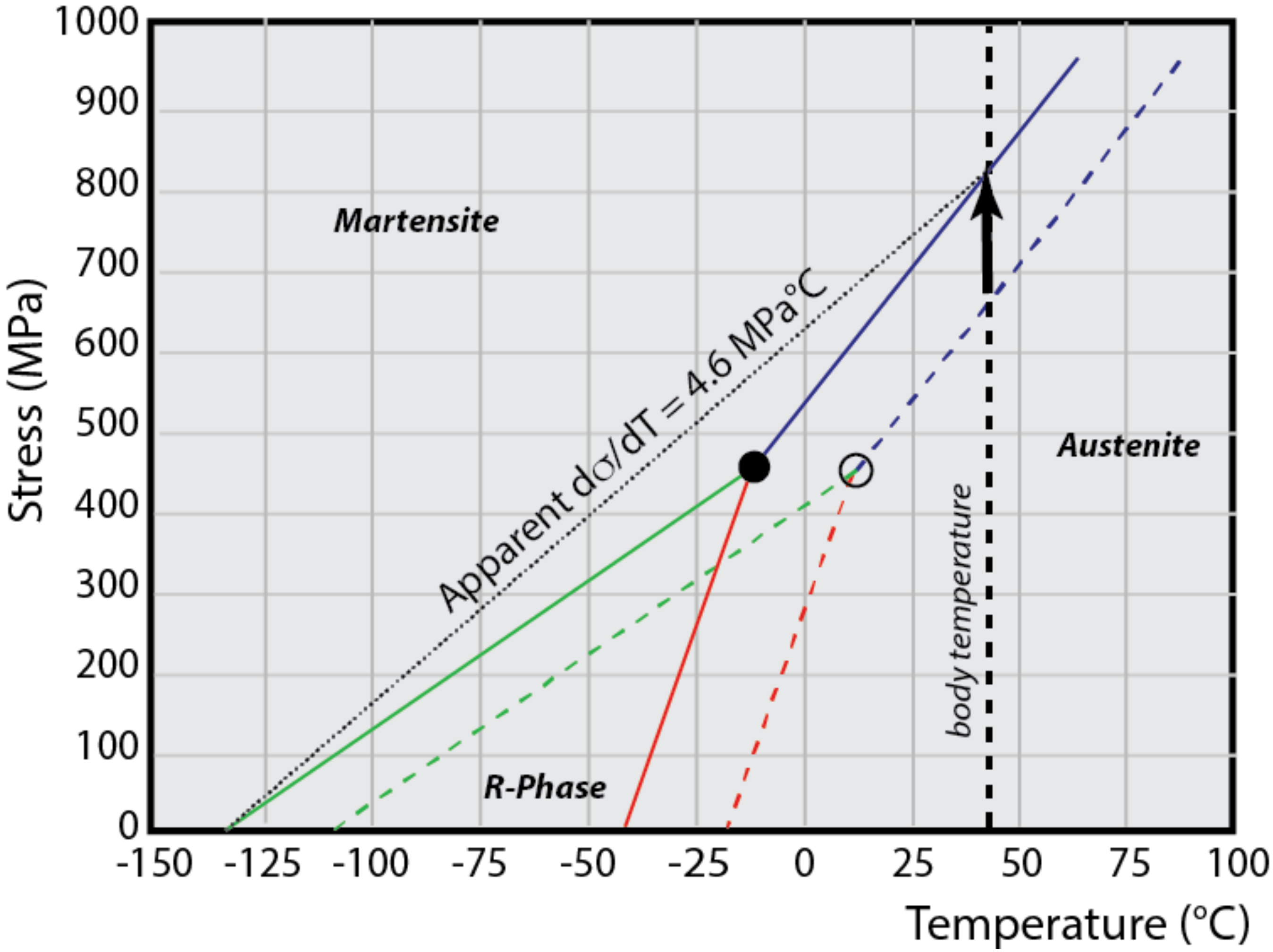
Forward diagram with triple point



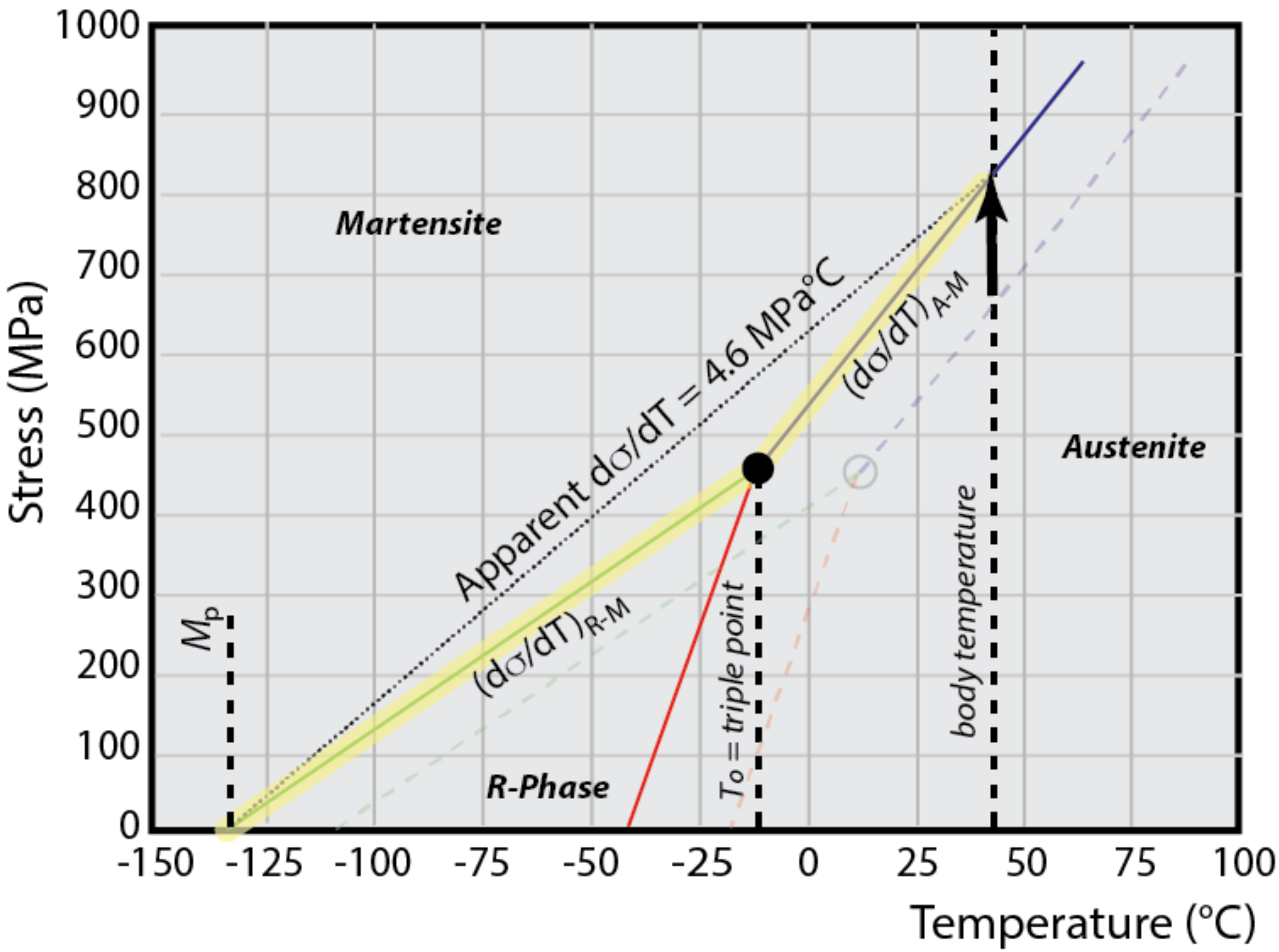
Shifting M down increases upper plateau stress



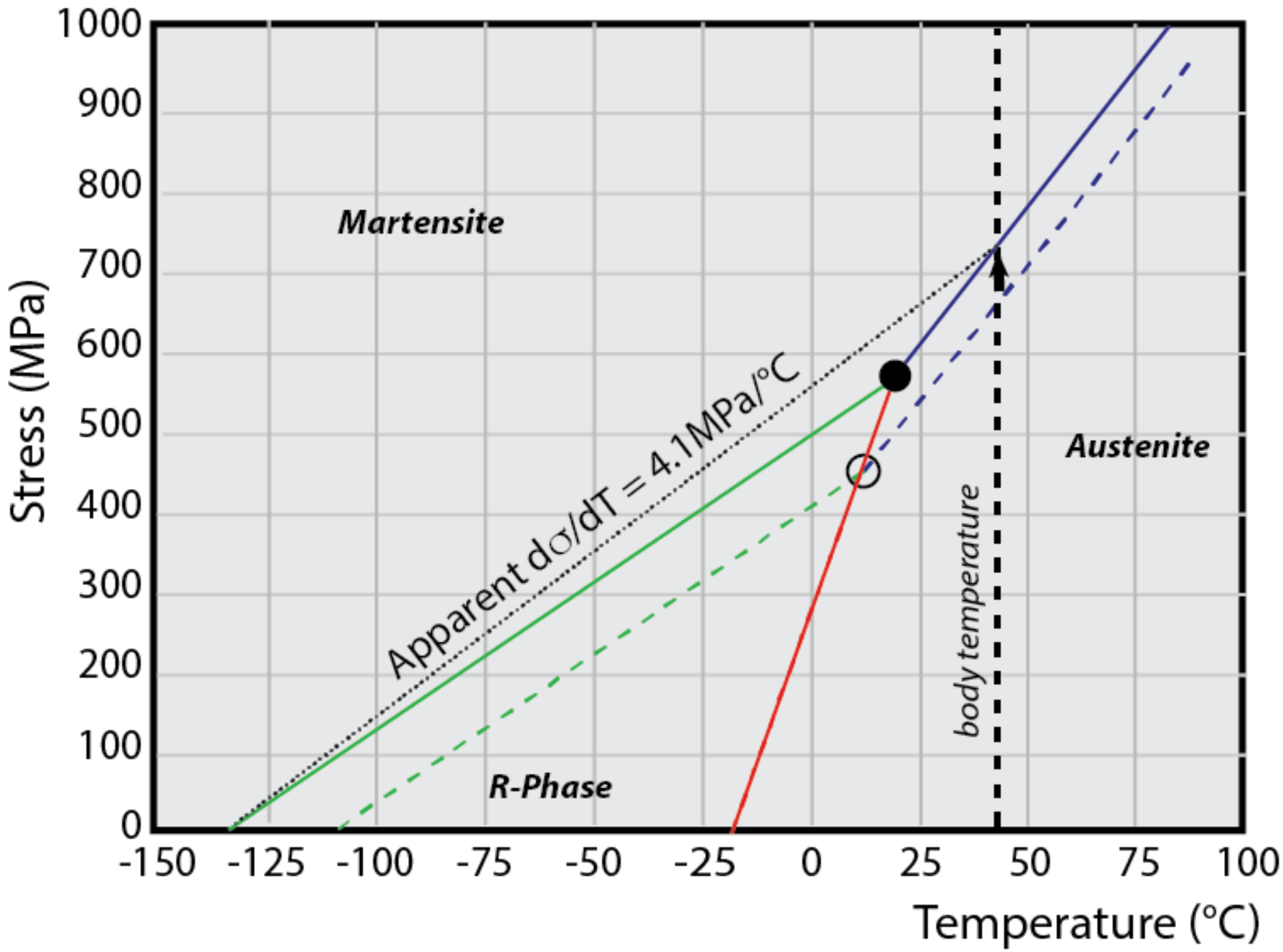
Apparent dσ/dT is an average weighted from the triple point



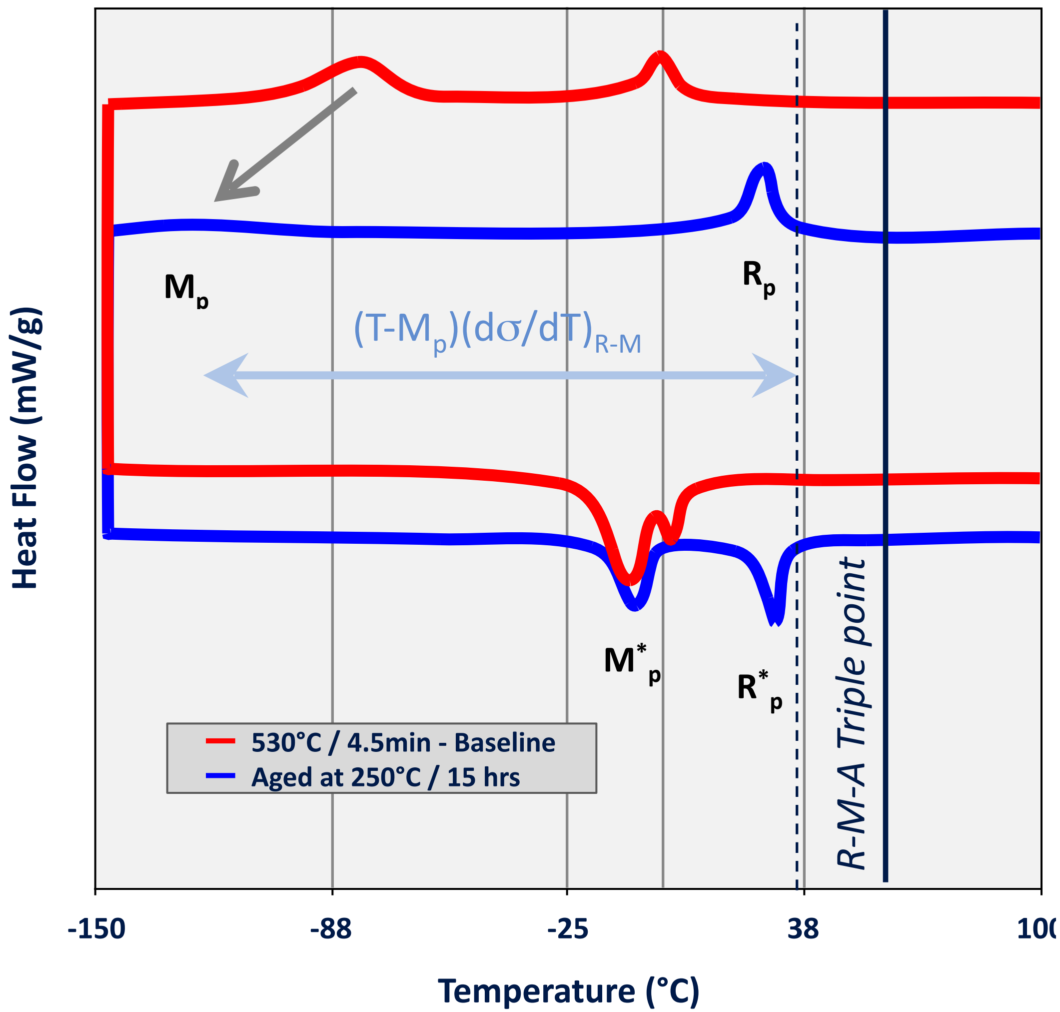
$$(d\sigma/dT)_{apparent} = [(T- T_o)(d\sigma/dT)_{A\text{-}M} + (T_o- M_p)(d\sigma/dT)_{R\text{-}M}] / (T - M_p)$$

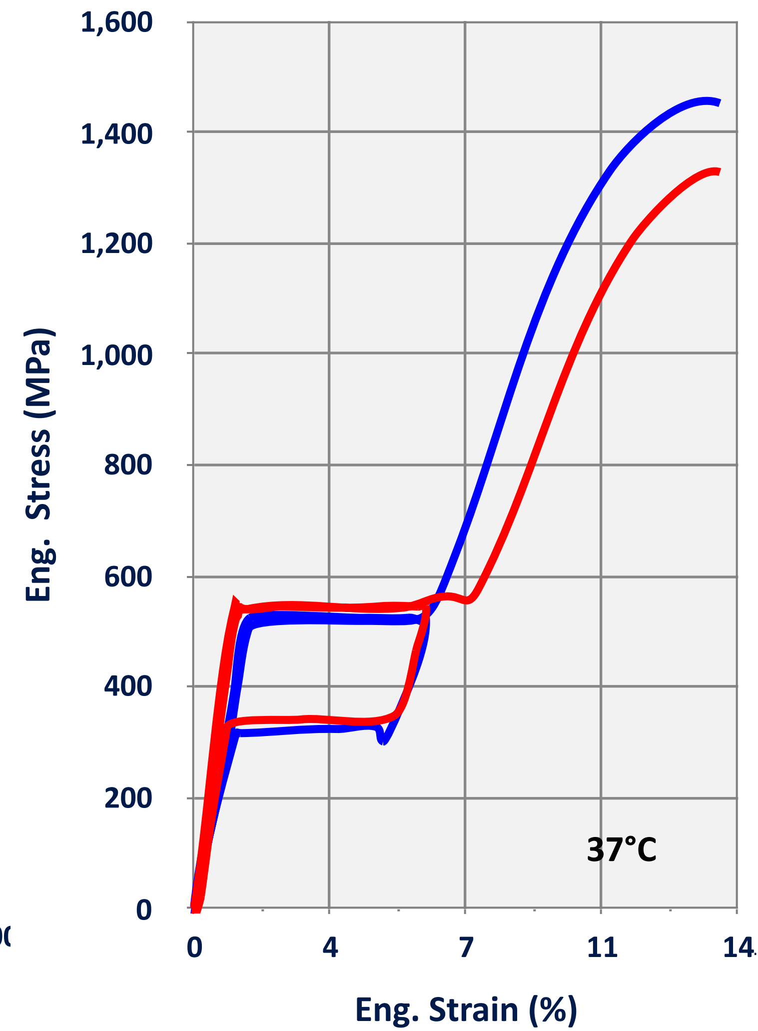


Shifting M without R, lowers apparent dσ/dT

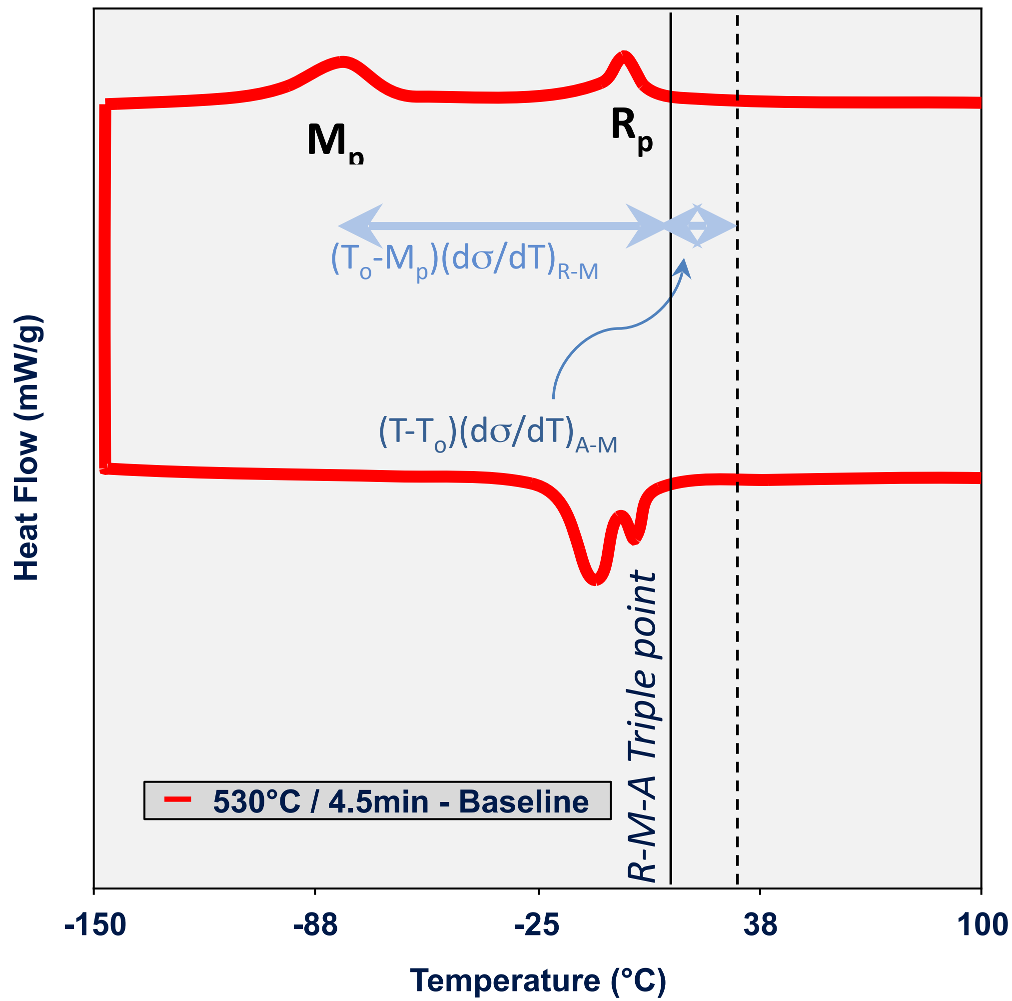


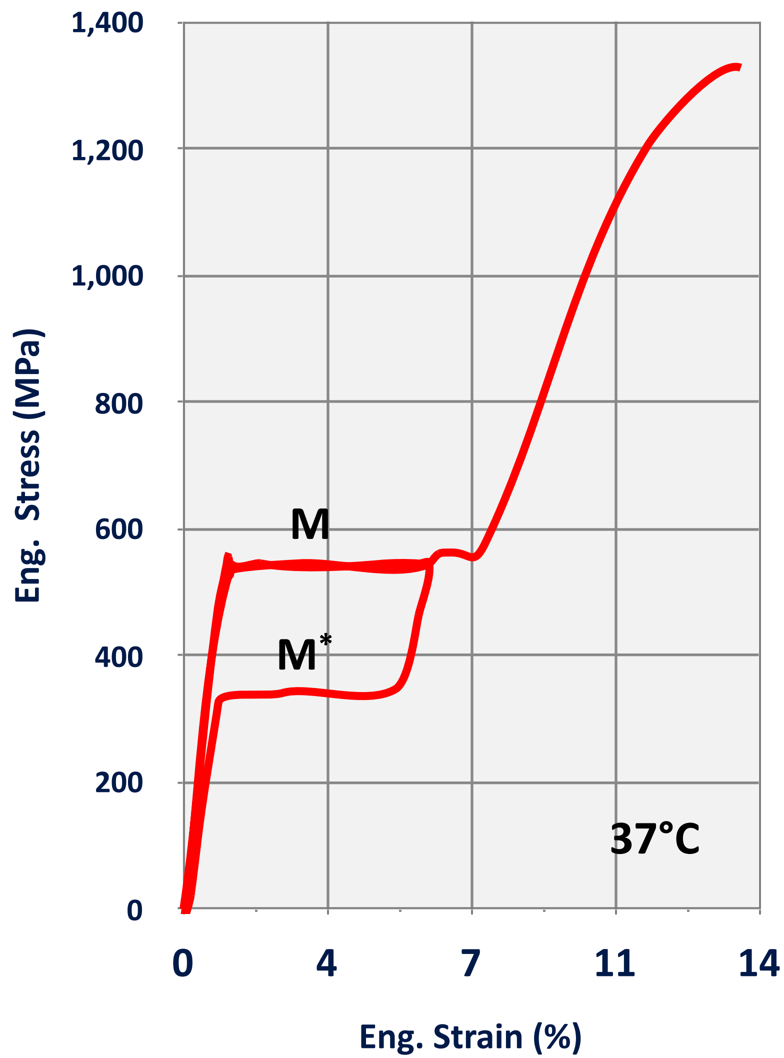
Low temperature aging decreases average dσ/dT
T is now below the triple point



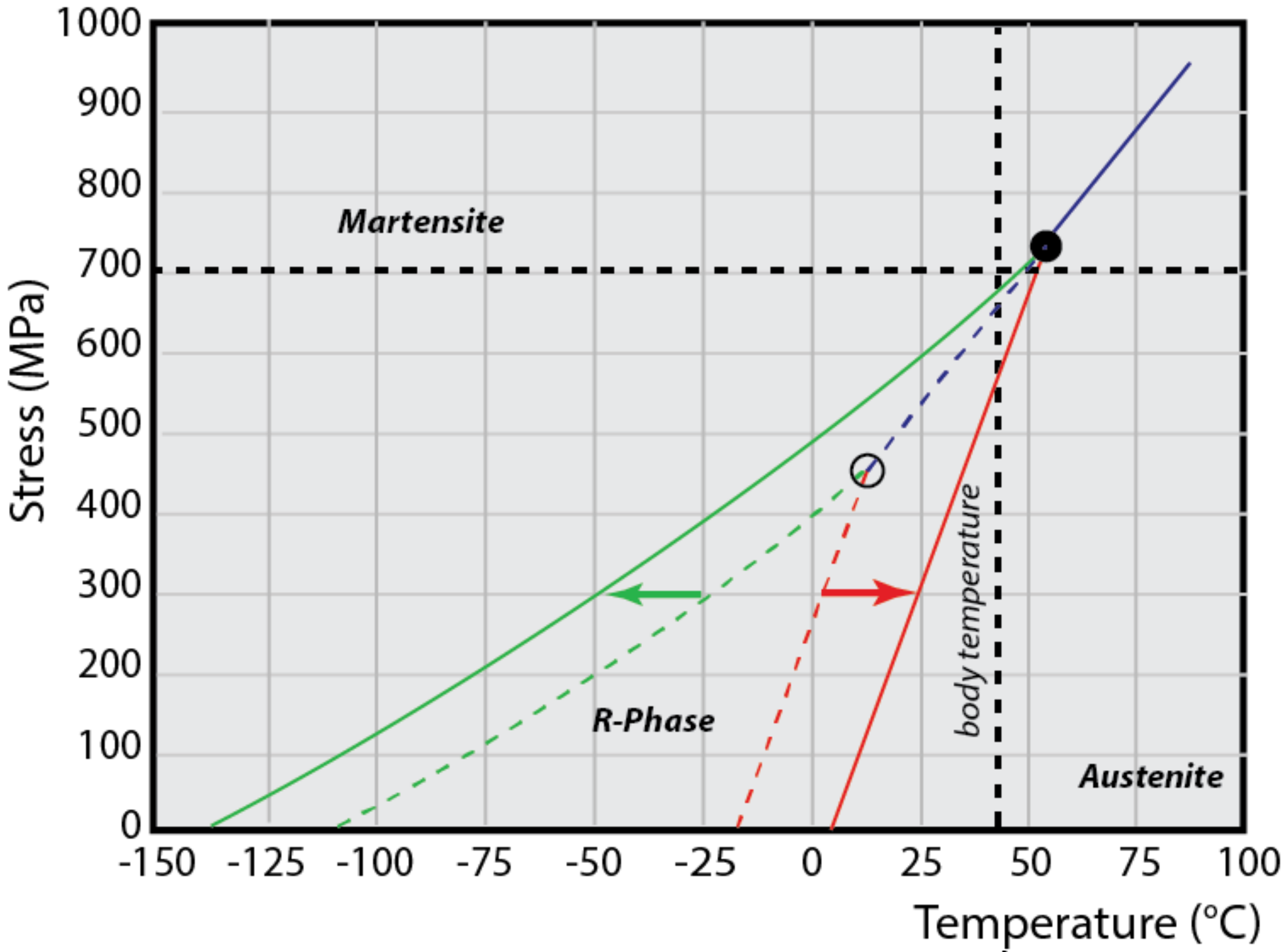


Traditional superelastic material





“Apparent” dS/dT depends upon how far ambient temperature is from the triple point



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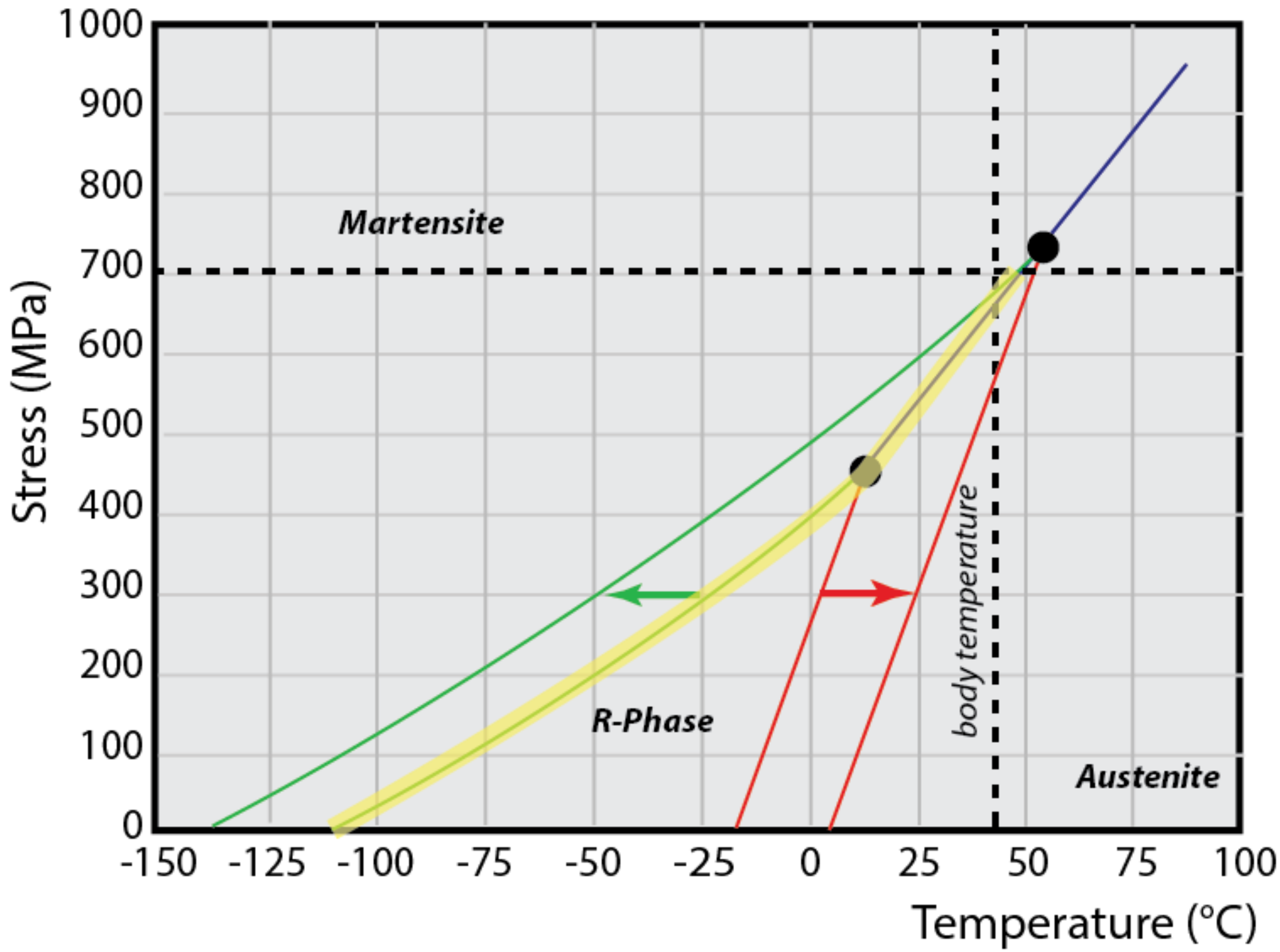

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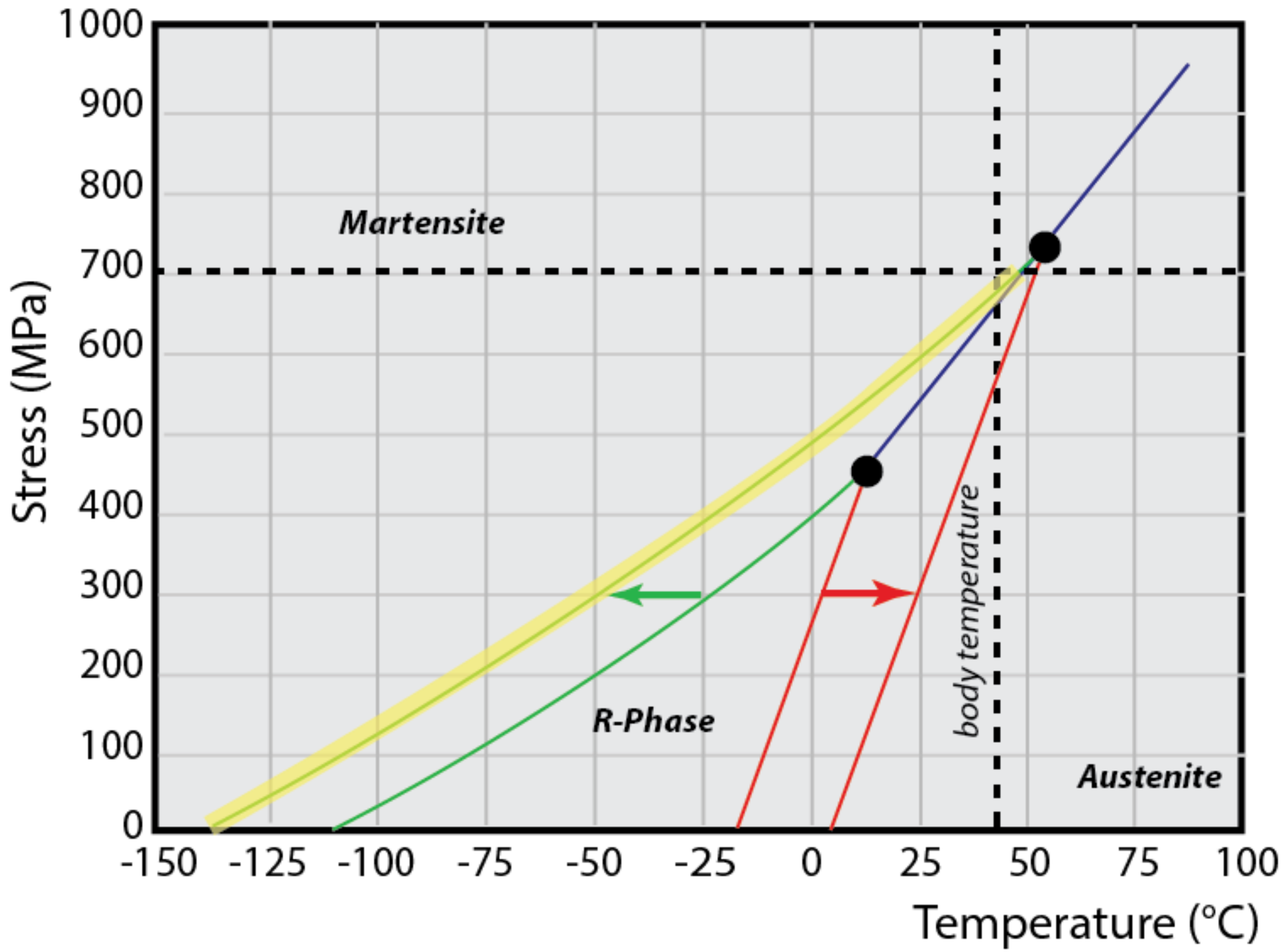
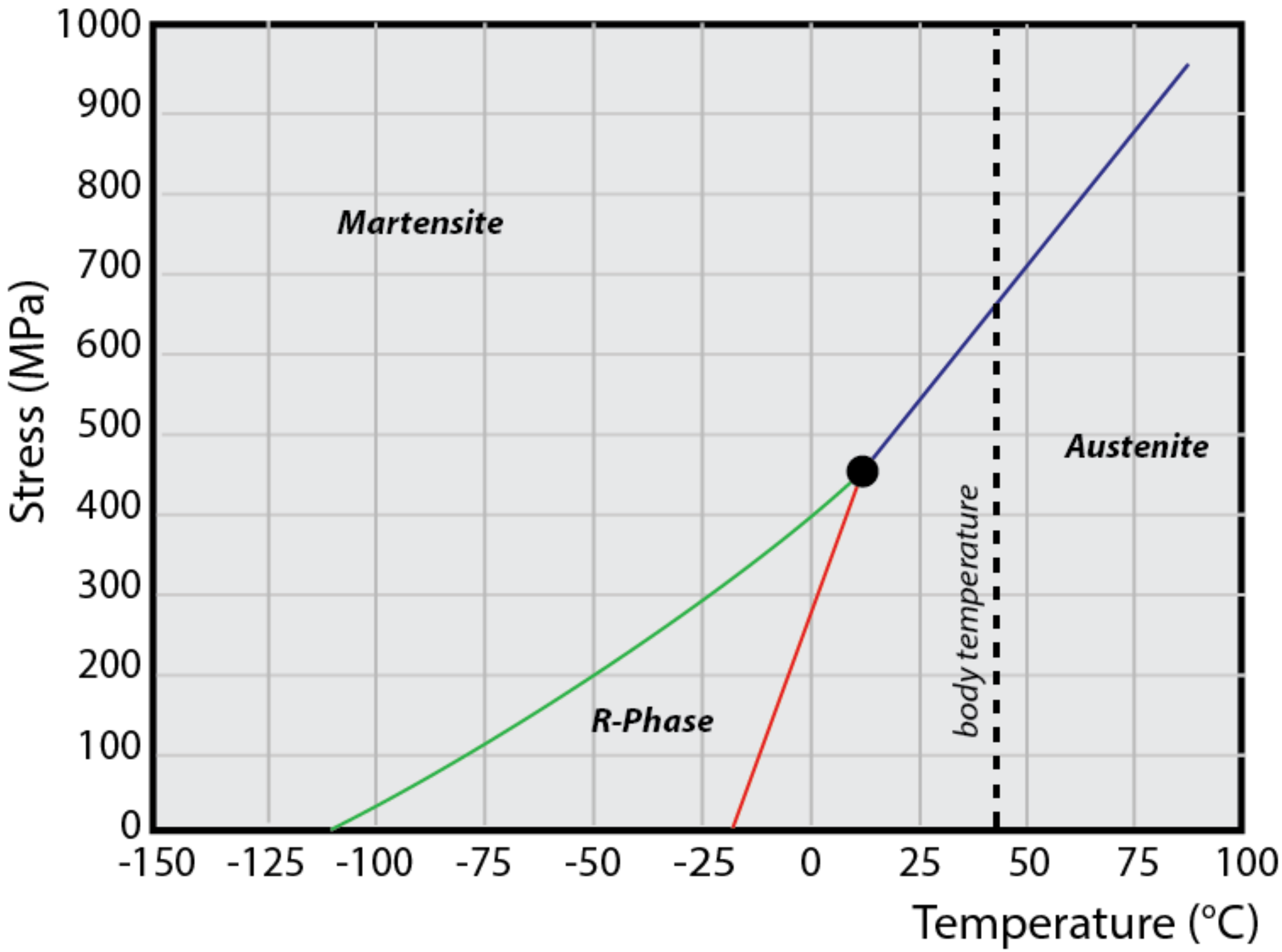


The R-M transus is non-linear

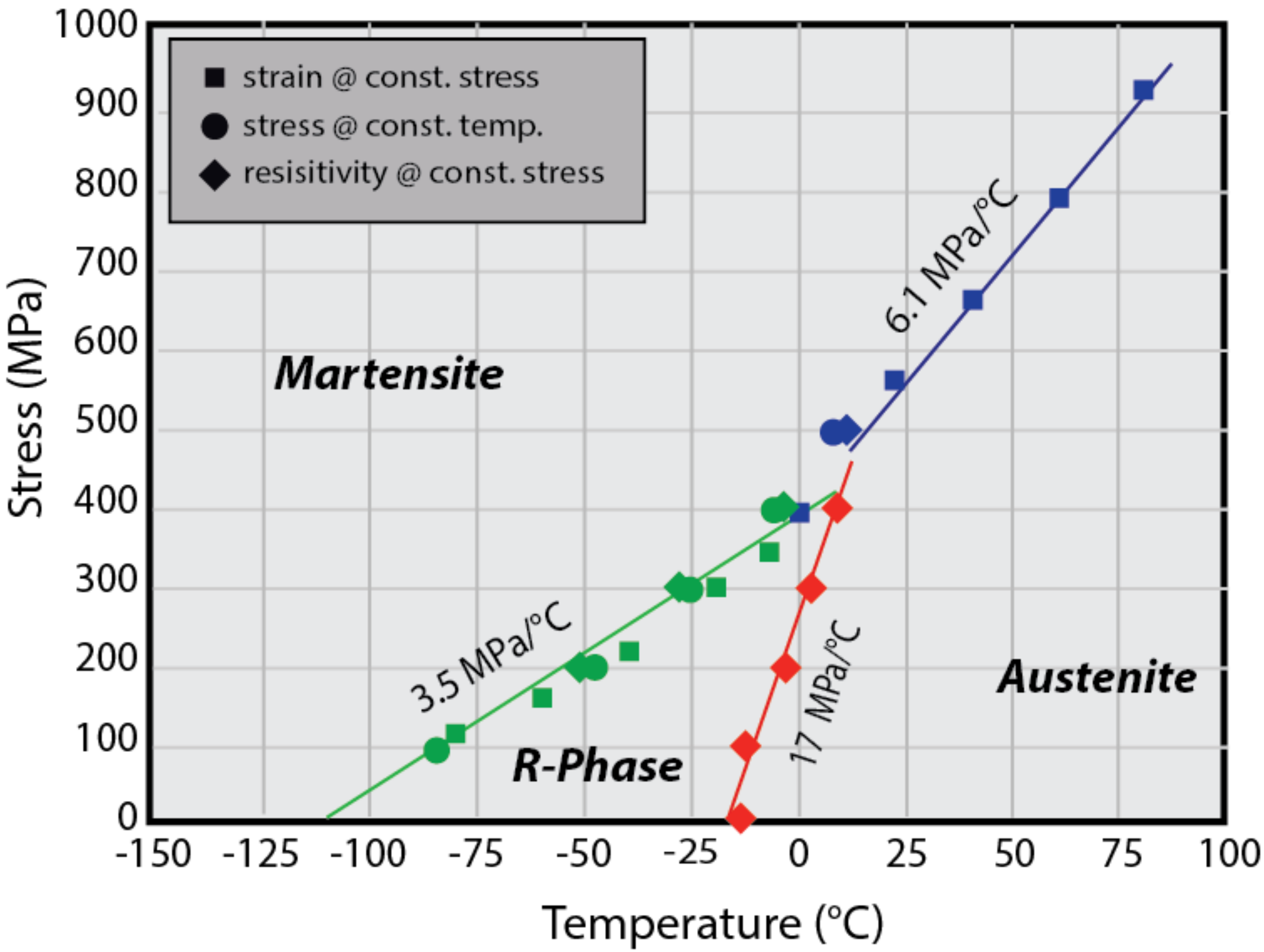
The R-phase rhombohedral angle contracts with continued cooling:

- ΔS decreases and
- $\Delta \varepsilon$ increases, so
- $d\sigma/dT$ contracts with temperature

The non-linear R-M transus



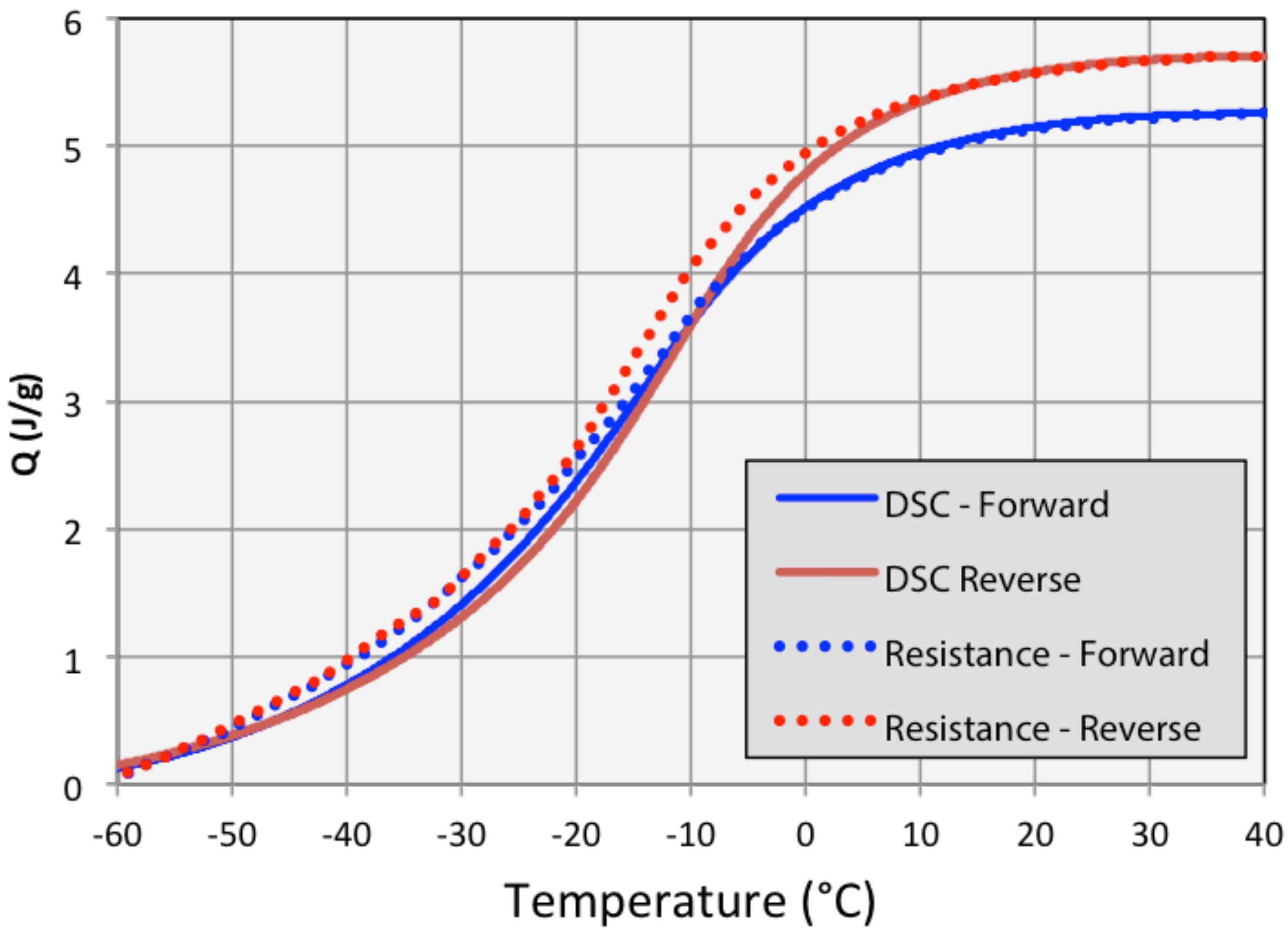
The full A-R-M phase diagram in the forward direction



CONFLUENT

Can one predict the transus slopes by measuring Q in a DSC?

Non-conservative contributions to Q prevent are minimal in the A-R transformation



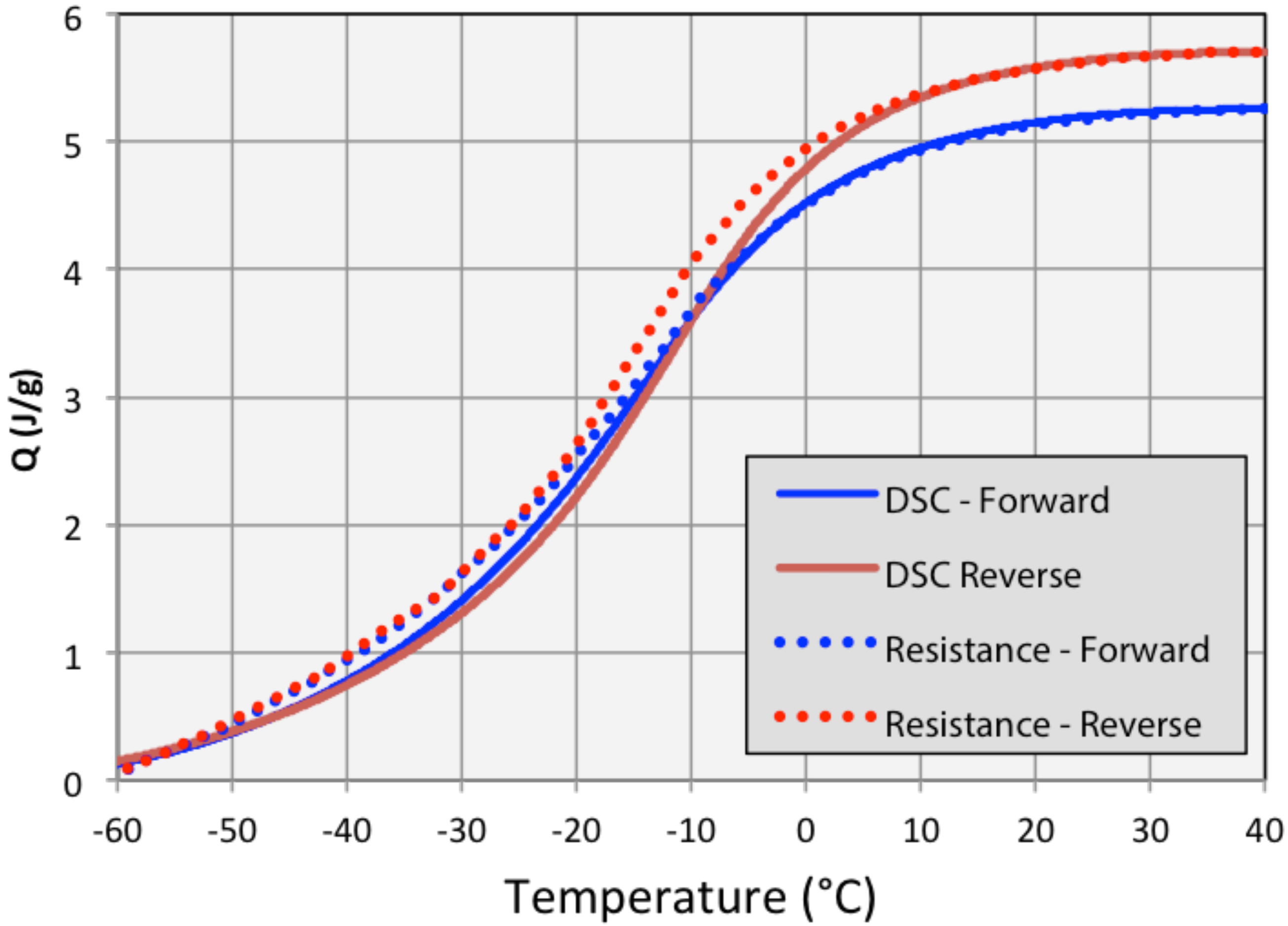
Calculating $d\sigma/dT$ from DSC fails even in the A-R case

Assuming $\Delta\varepsilon$ of 0.5% and $\Delta H \sim Q = 5$ J/g:

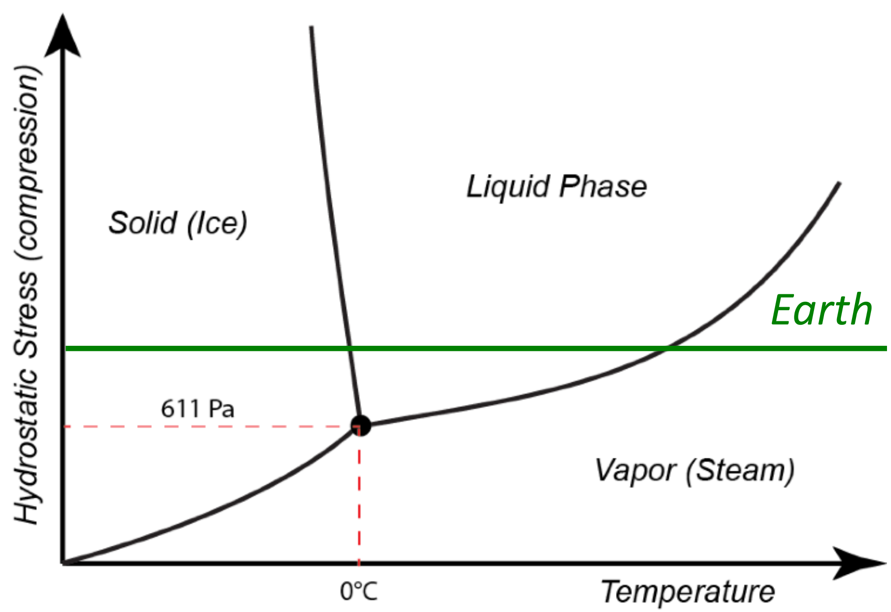
$(d\sigma/dT)_{A\text{-}R} = \Delta H/T\Delta\varepsilon = 29$ MPa/°C (versus 17 MPa actual)

CONFLUENT

But if you really want a fast, non-destructive, accurate way to measure the true A_f, use resistivity.



- A more useful and less ambiguous terminology has been proposed: M* and R*
- A phase diagram was established for commercially relevant superelastic wire in tension
- The diagram is well predicted by DSC



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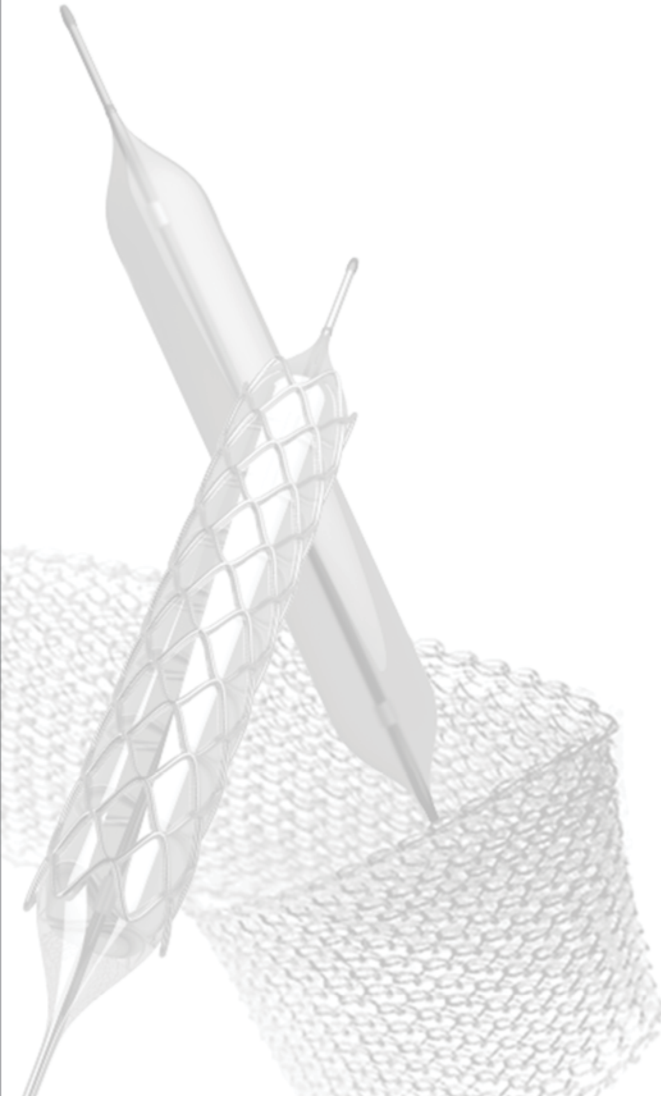

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