# “Reference QuenchProbe" – An Answer to all your Queries on Quenchants

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TherMet Solutions
Private Limited
Incubated at IIT Madras INDIA

# “Reference QuenchProbe" – An Answer to all your Queries on Quenchants

- Understanding your quenchant properties
- Developing process control charts
- Optimizing the required parameters
- Freeze the final parameters for optimal results

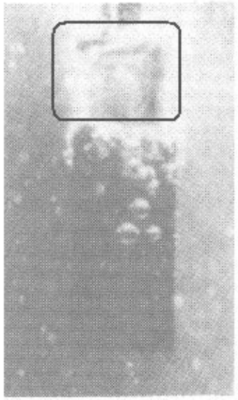
# Immersion Quenching – An Introduction

- Immersion Quenching is accompanied by boiling / vaporization of the fluid medium - one of the most complicated phenomenon to quantify

- Heat transfer during quenching of steel occurs in several stages
  - Vapor phase – a thin blanket of fluid vapor separates the component surface and the quenchant
  - Nucleate boiling – the vapor blanket breaks down and the fluid comes in direct contact with the component surface
  - Convective heat transfer –boiling ceases and the heat transfer is through the convective mechanism

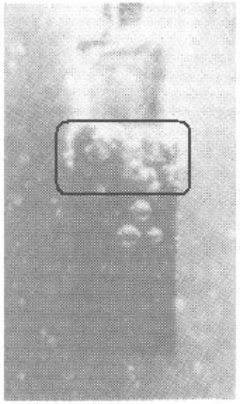
- Every point on the component surface goes through the three stages, each stage lasting for different durations

# Heat Transfer during Quenching*



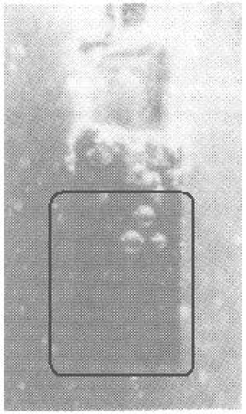
10.0 s

Film boiling phase: Heat transfer impeded by a vapor blanket



10.0 s

Nucleate boiling phase: Maximum heat transfer due to wetting

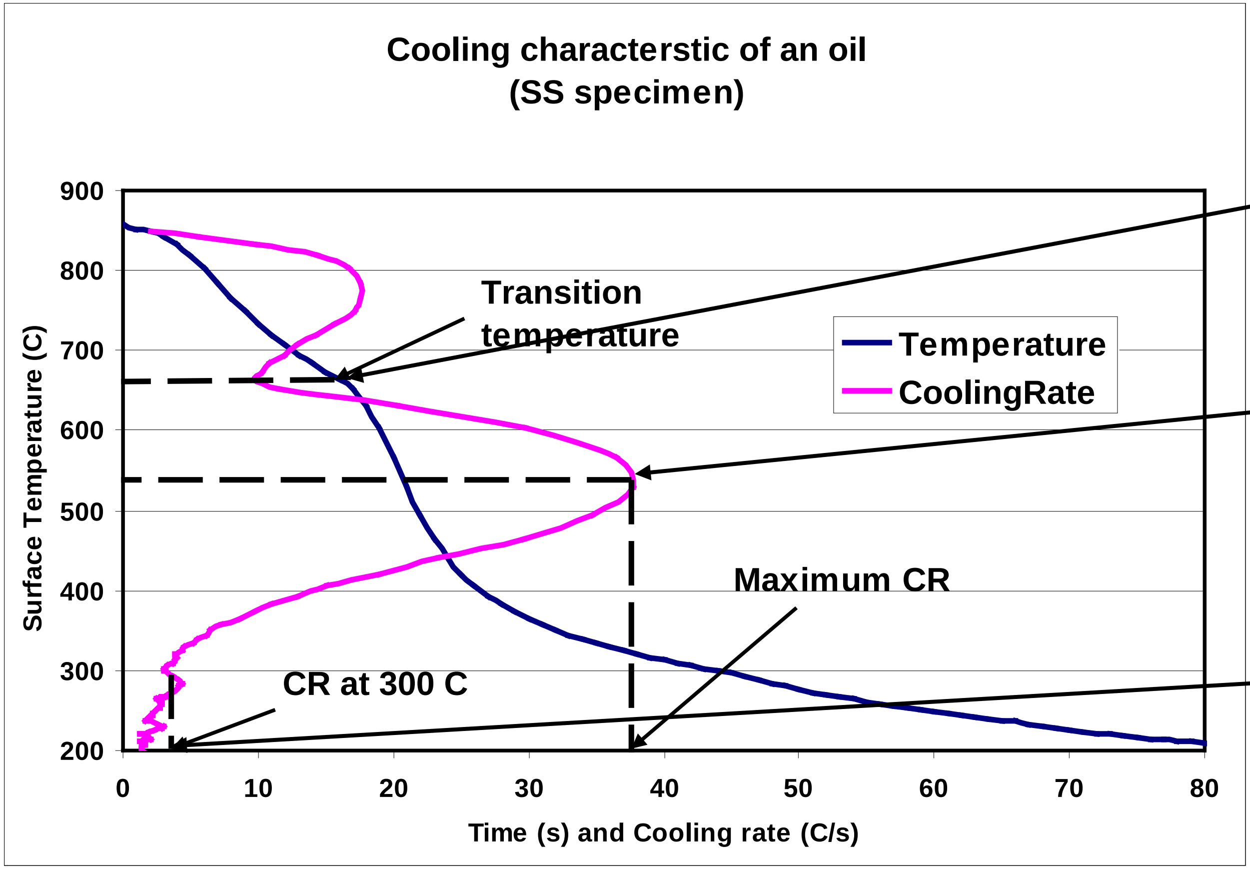


10.0 s

Convective Phase: Low heat transfer through convection

***"Handbook of Quenchants and Quenching Technology," 1993, Eds: G.E.Totten, C.E.Bates, N.A.Clinton, ASM**

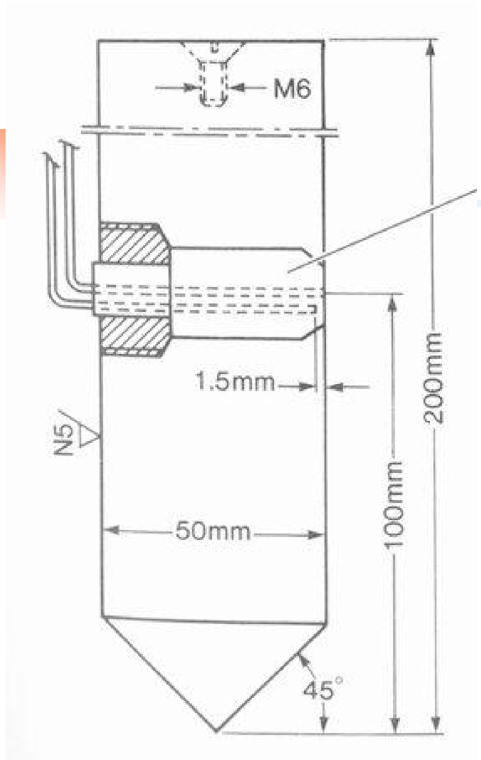
# Analysis of Cooling Curves



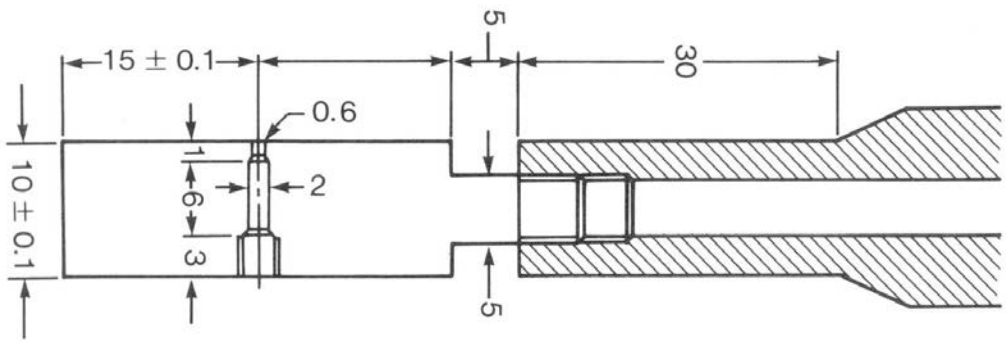

Vapor blanket to film boiling

Must be sufficiently high to minimize ferrite and pearlite transformation

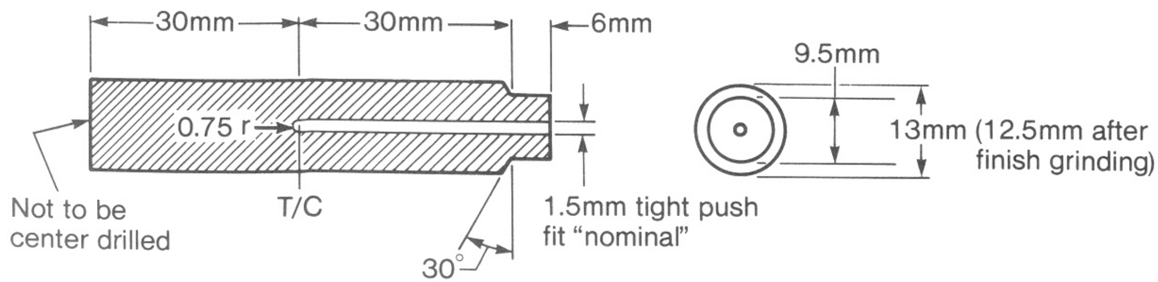
Indicates probability of distortion and cracking



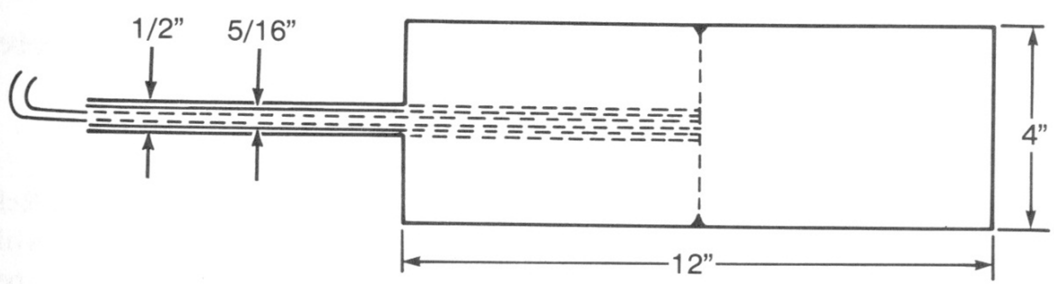

Liscic NANMAC Probe for measuring heat flux – Austenitic steel specimen




Silver probe for surface temperature measurement




Wolfson Inconel probe (Single cooling curve)




The Grossman probe –SAE 5145 (Single cooling curve)

# Obtaining Cooling Curves - Conventional Probes - Limitations

- Grossman probe, JIS Silver probe, French Probe, Wolfson Inconel probe, Cylindrical Silver Probe, Allen Plate Probe

- How to interpret the results – material is different – test conditions are different

- What does a change in the cooling curve actually mean – how to map it on to my material and my plant conditions?

- Should one continue with the existing bath, top it up or replace – what should be the basis?

# The ASTM Standards for Quenchant Testing - D6200-01(2012)

## Interpretation of Results

- Cooling curves and cooling rate curves are obtained for
  - Comparing one oil with other
  - Comparing one oil with control sample
  - Compare with previous performances

- The test may show the effect of
  - Oxidation
  - Presence of additives
  - Contamination

  on the cooling characteristics of a quenching oil.

# Standard parameters for testing quenching oil as per ASTM

- Viscosity
- Specific Gravity
- Contamination
- Water Content
- Flash Point

| Parameter | Min. % of Ref. | Max % of Ref. |
|---|---|---|
| Max Cooling Rate C/sec | 90 | 110 |
| Temp @ Max Cooling Rate C | 95 | 105 |
| Cooling Rate @ 300 C C/sec | 70 | 130 |
| Time to Reach 600 C sec | 85 | 115 |
| Time to Reach 400 C sec | 90 | 110 |
| Time to Reach 200 C sec | 90 | 110 |

# The ASTM Standards for Quenchant Testing - D6200-01(2012)

**Limitations**

- Designed to evaluate quenching oils in a non-agitated system.
- No correlation between these test results and the results obtained in agitated systems.
- No correlation between the test results and results with different grades of steel

# Parameters affecting Cooling Curves during quenching

- Cooling curves are affected by bath conditions
  - Bath temperature, Agitation, Quenchant type, Section size, Steel Composition

- Cooling curves are also affected by bath degradation
  - Continuous usage of oil results in oxidation and
  - Contamination of oil with water, polymer etc.
  - Concentration of Polymer
  - The concentration and temperature of NaCl can affect the quenchant characteristics

# Addressing the limitations of the standard probes using ReferenceProbe

- Specimen material same as of component to be taken
- Fix thermocouple at 4mm below surface
- Heat to soaking temperature
- Quench in plant conditions

# Features of “Reference QuenchProbe”

- Cylindrical specimen (25 dia x 100 long) -
  - Same grade as component: For material-quenchant-plant specific cooling curves
- Single temperature measured at 4mm from the surface; Mineral insulated Inconel sheathed ‘K’ type thermocouple
- Patented design for fixing the thermocouple for positive contact
- Unique USB based data logger for portability; Integrated design
- Portable electric resistance furnace for heating the specimen



Integrated design; the handle with a view port receives the data logger which is thus physically protected for plant use; body fabricated of stainless steel with bright finish; weight of the probe finely balanced

# Hardening Power – Grossman Number

- Grossman number - a measure of cooling capacity of quenchant
  *H* = htc/2*(th.cond)

- htc varies continuously during quenching

- Single value of H cannot be attributed to a steel and quenchant combination

- Still, widely accepted by heat treaters:

| | |
|---|---|
| – Slow oil – no agitation | 0.20 |
| – Good oil – moderate agitation | 0.35 |
| – Poor water – no agitation | 1.00 |
| – Very good water – strong agitation | 1.50 |
| – Brine – no agitation | 2.00 |
| – Brine – violent agitation | 5.00 |

# Hardening Power - Heat Flux Rate

- Liscic Probe

- Heat flux at surface measured by two TCs by gradient method
  - Assumes linear dT/dx
  - Actual surface temperature cannot be measured directly as this method claims

- Austenitic stainless steel probe
  - Results cannot be applied to other materials

- Microstructure and hardness in alloy steel cannot be computed based on SS heat flux

# To summarize …..

Acquisition of cooling curve and subsequent analysis should ensure:

- Specificity of material – quenchant combination
- Tests done under plant conditions – to replicate practical issues
- Use experimental data (TTT and hardness data)
- Develop mathematical models to relate plant conditions to data base
- Easy and hassle free test procedure

**The new "Reference QuenchProbe" addresses these issues**

# Theory of "QuenchProbe" - TTT (IT) Diagrams

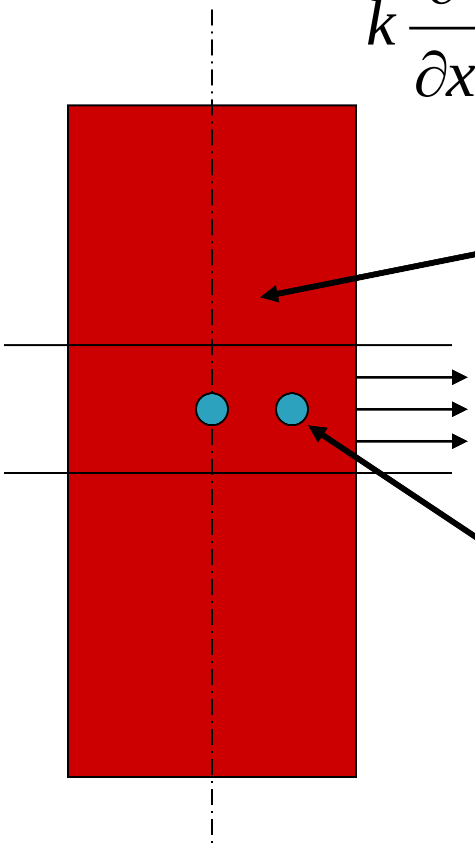
- Give information about the product phases (ferrite, pearlite, bainite, martensite, austenite); sometimes, the hardness of the resulting phases also is given
- Steel component is held at the temperature of transformation for long enough times – not cooled !
- They are 'read' only along the isotherms
- Very limited direct application
- Interpreted through mathematical models

# Theory of “QuenchProbe” - CCT Diagrams

- Give the information of transformation products when a steel is cooled continuously at variable cooling rates
- Many ways of construction
  - Based on following different cooling paths
  - Based on cooling rates at a temperature
  - Based on different diameter specimens in different quenchants
  - Based on time to cool between two temperatures
- Used as guidelines by superimposition
- Proper interpretation through mathematical modeling

# Theory of "QuenchProbe" – Inverse Heat Transfer

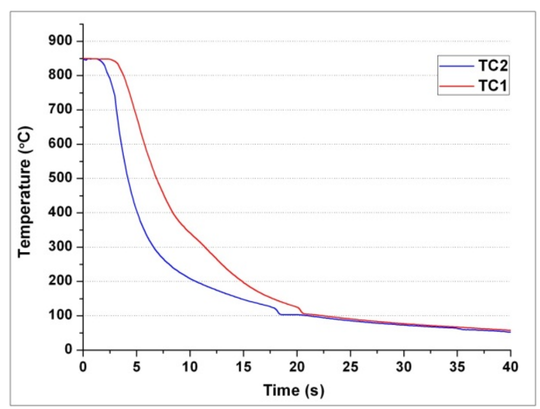
$$k\frac{\partial}{\partial x}\left(\frac{\partial T(x,y,t)}{\partial x}\right)+k\frac{\partial}{\partial y}\left(\frac{\partial T(x,y,t)}{\partial y}\right)+\dot{q}=\rho c\frac{\partial T(x,y,t)}{\partial t}$$



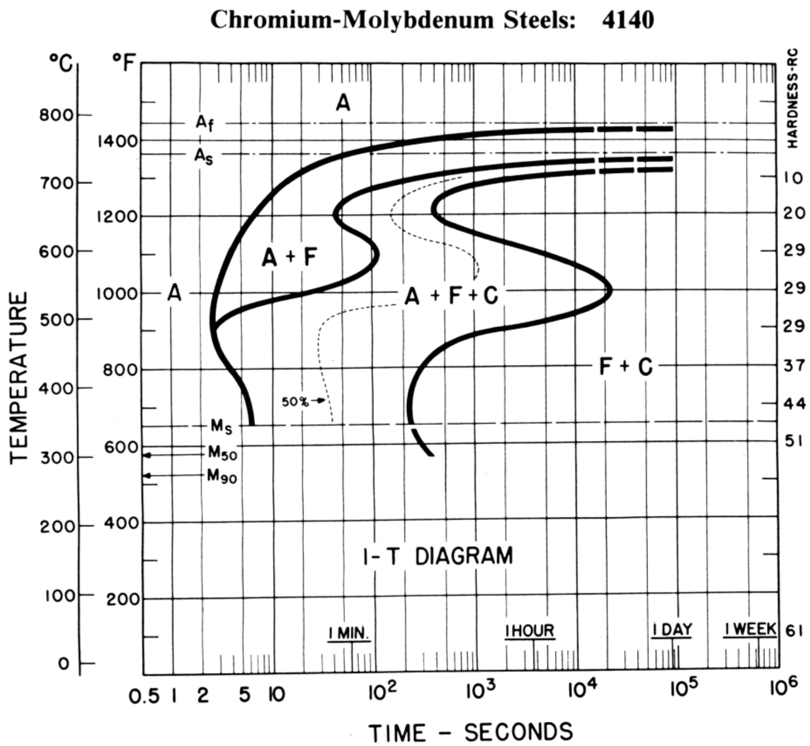
IC: $$T(x,y)=T_{soaking}$$

BC: $$-k\left(\frac{\partial T}{\partial x}n_x+\frac{\partial T}{\partial y}n_y\right)=q=?$$

Input:



# Theory of "QuenchProbe" – Metallurgical Model




Austenite – Ferrite/Pearlite/Bainite

$$X_i^{\,j} = 1 - \exp[-b(T_j)\, t_j^{\,n(T_j)}]$$

$$n(T_j) = \frac{\ln[\ln(1-X_s)/\ln(1-X_f)]}{\ln(\tau_s/\tau_f)}$$

$$b(T_j) = -\frac{\ln(1-X_s)}{t_s^{\,n}}$$

Austenite - Martensite

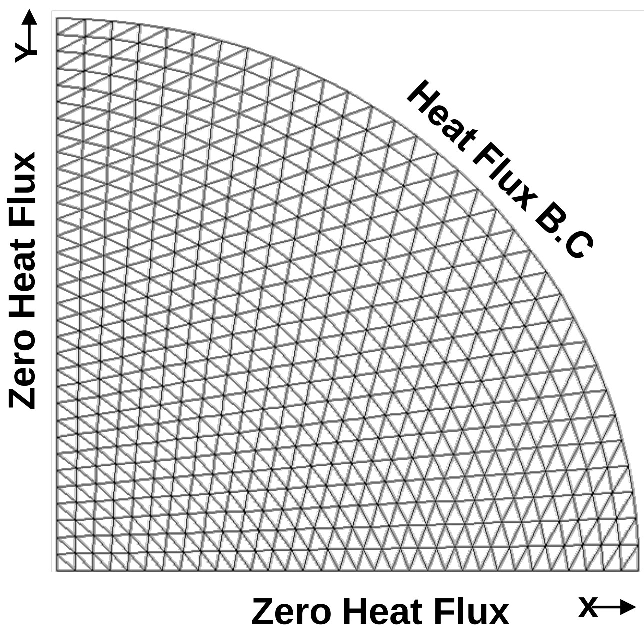
$$X_m = 1 - \exp[-0.011(M_s - T)]$$

# Theory of “QuenchProbe” – Finite Element Analysis

Iterative FE formulation within time step for non linear problems:

$$\left(\Delta t\theta[K]_{n+1}^{l+1}+[C]_n\right)\{T\}_{n+1}=\left([C]_n-\Delta t(1-\theta)[K]_n\right)\{T\}_n+\Delta t(1-\theta)\{F\}_n+\Delta t\theta\{F\}_{n+1}^{l+1}$$

$$C_{ij}=\int_{\Omega^e}\left[\rho c-\rho\Delta H\left(\frac{\Delta f}{\Delta T}\right)\right]\psi_i\psi_j\,d\Omega \qquad K_{ij}=\int_{\Omega^e}\left[k\frac{\partial\psi_i}{\partial x}\frac{\partial\psi_j}{\partial x}+k\frac{\partial\psi_i}{\partial y}\frac{\partial\psi_j}{\partial y}\right]d\Omega \qquad F_i=-\oint_{\Gamma_2}q\psi_i\,d\Gamma$$




Objective function: $$S=\sum_{i=1}^{r}\left(Y_{m+i-1}-\hat{T}^{+}{}_{m+i-1}\right)^2$$

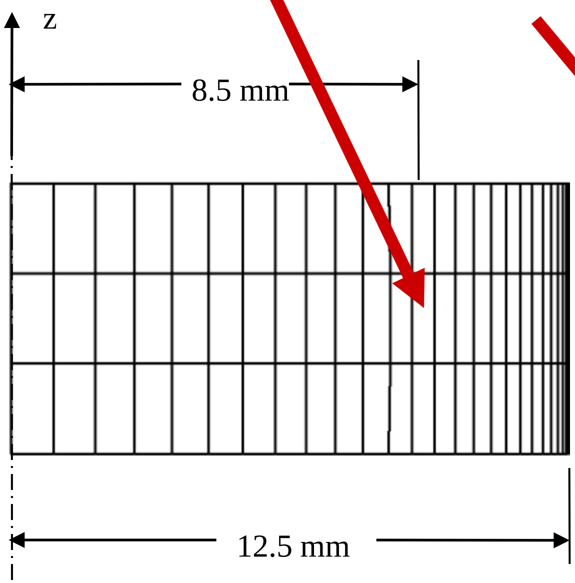
Flux computed from: $$(\Delta q)_m=\frac{\sum_{i=1}^{r}\left[Y_{m+i-1}-\hat{T}_{m+i-1}\right]\phi_i}{\sum_{i=1}^{r}(\phi_i)^2}$$

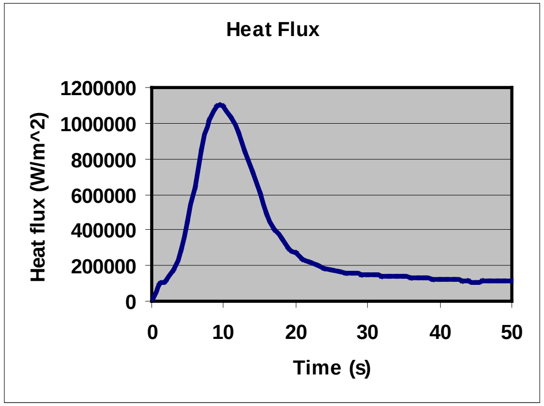
Using Sensitivity Coefficient: $$\phi_i=\frac{\left(\hat{T}^{+}{}_i-\hat{T}_i\right)}{\Delta q_i}$$

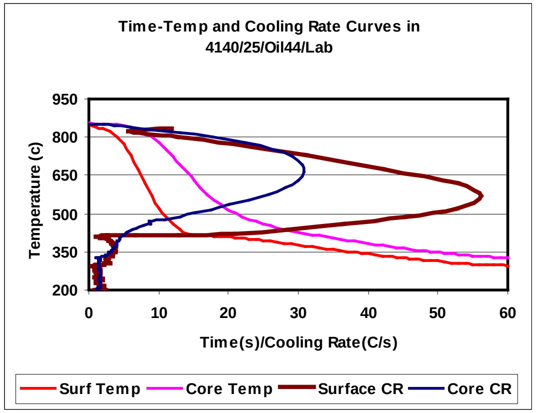
# Coupled Mathematical Modeling – Input/Output

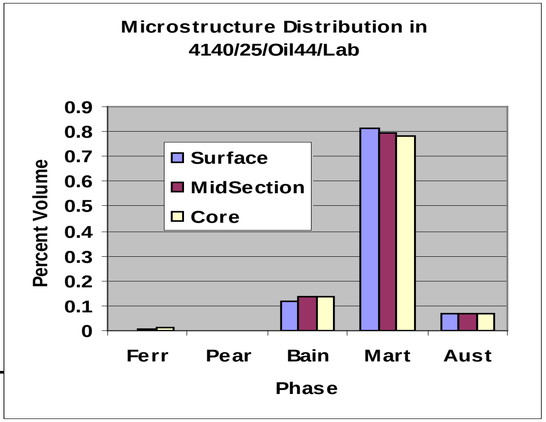
With a single measurement here

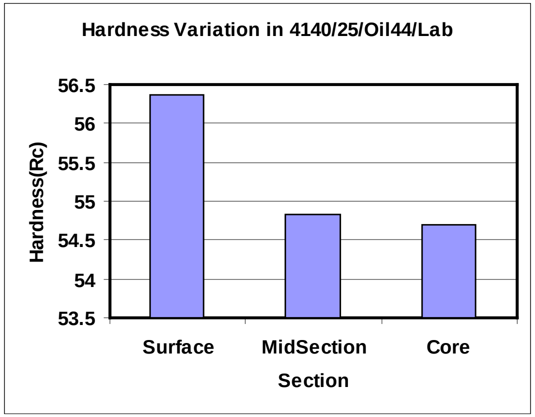
We get



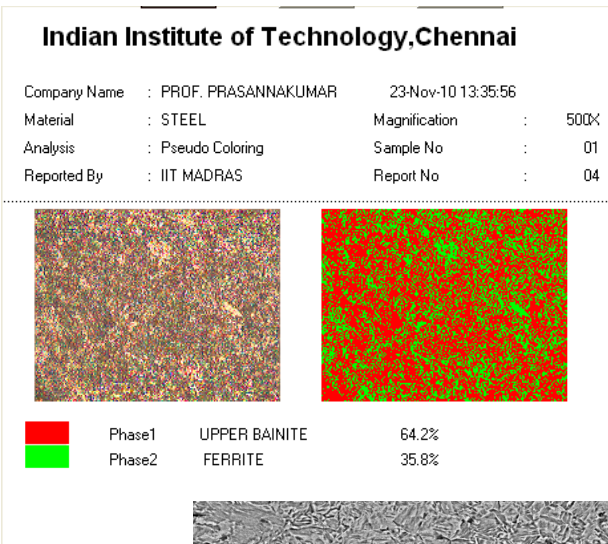



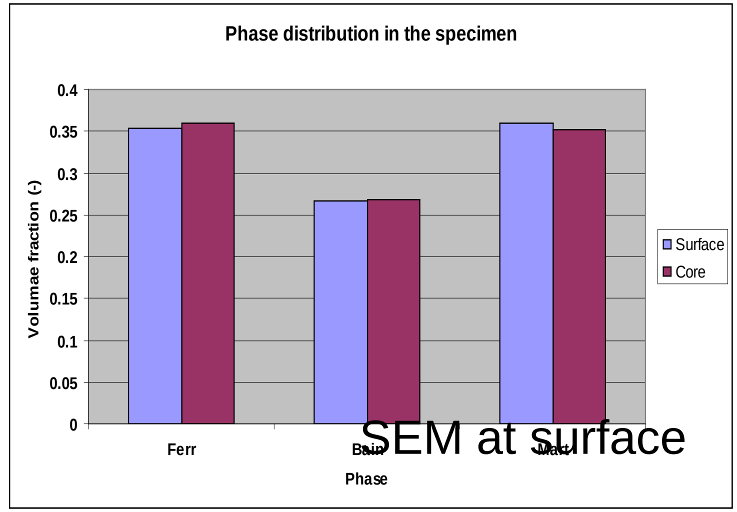



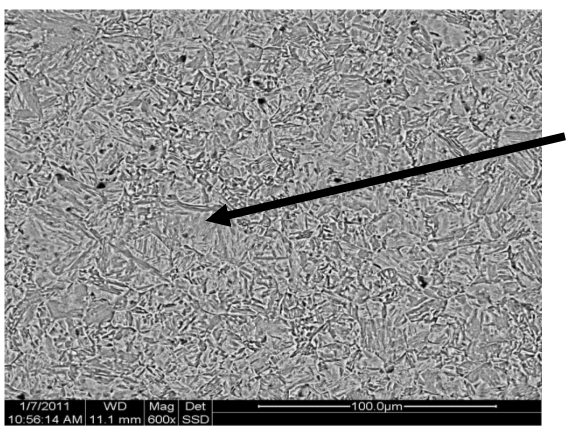



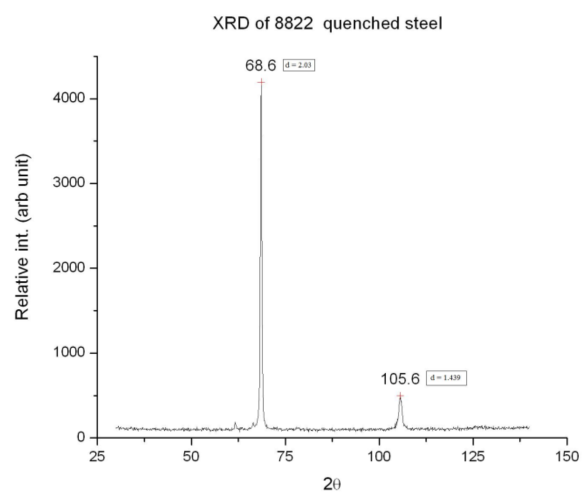
# Validation –Oil Quenched – AISI 8822H



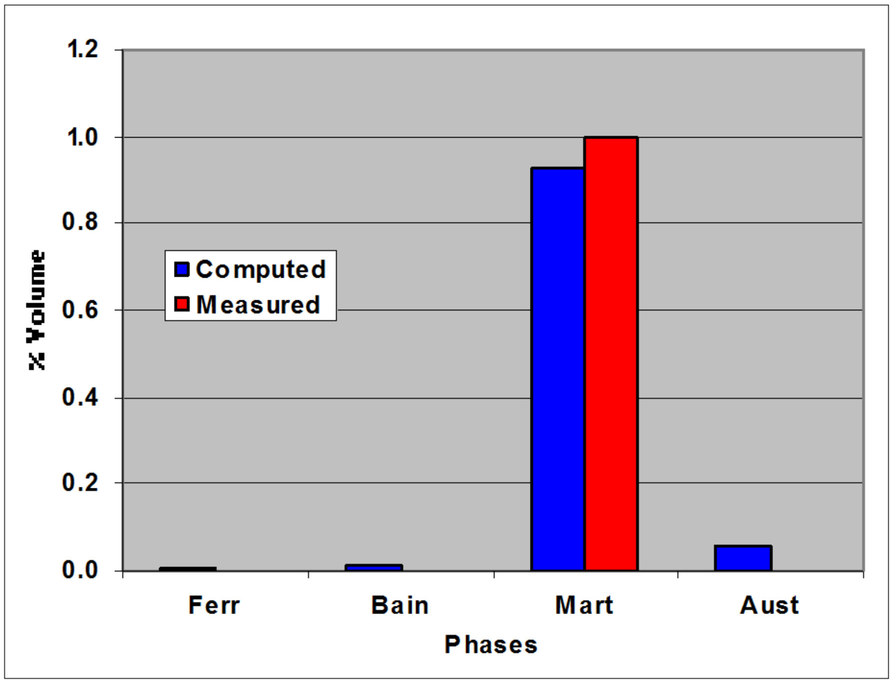



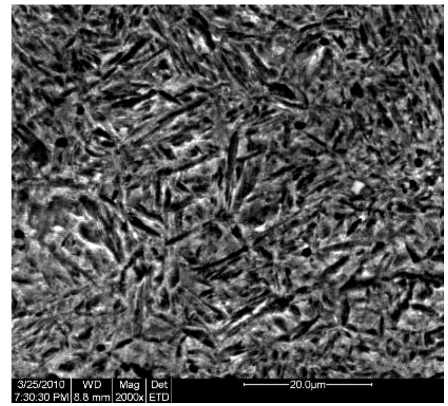

SEM at surface



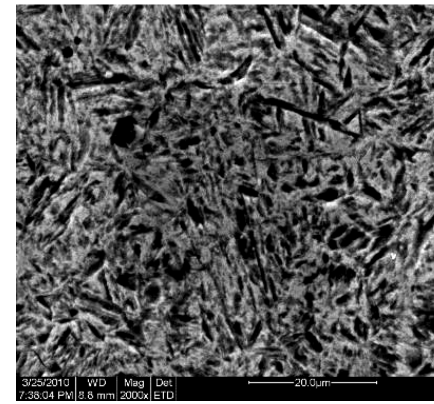



# Validation –Water Quenched – AISI 4140

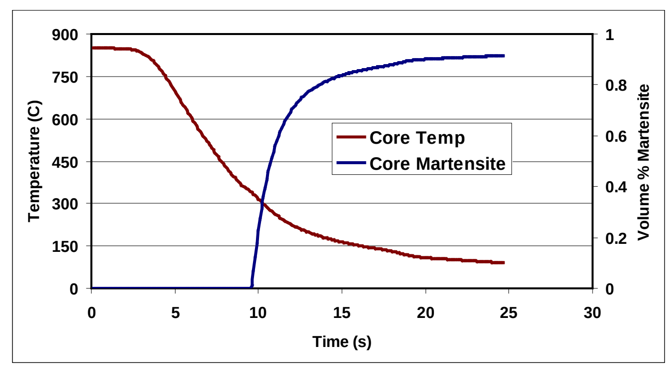


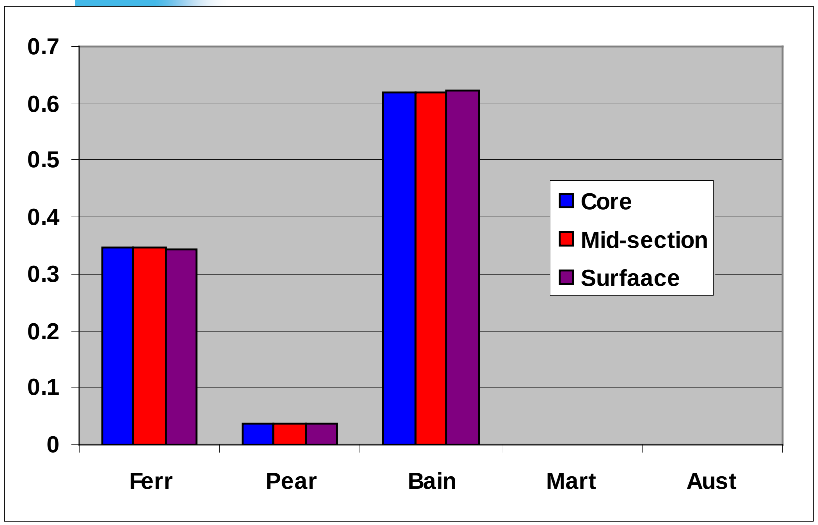
SEM at surface

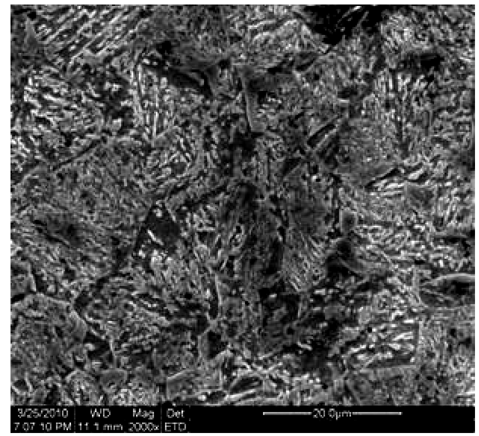


SEM at Core

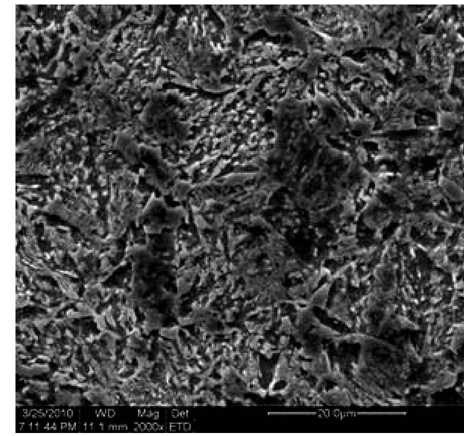


# Validation -Air Cooled - 4140

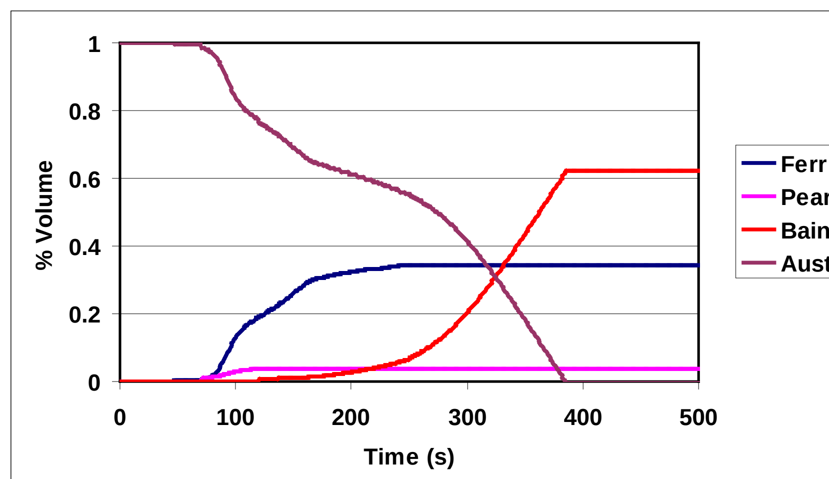


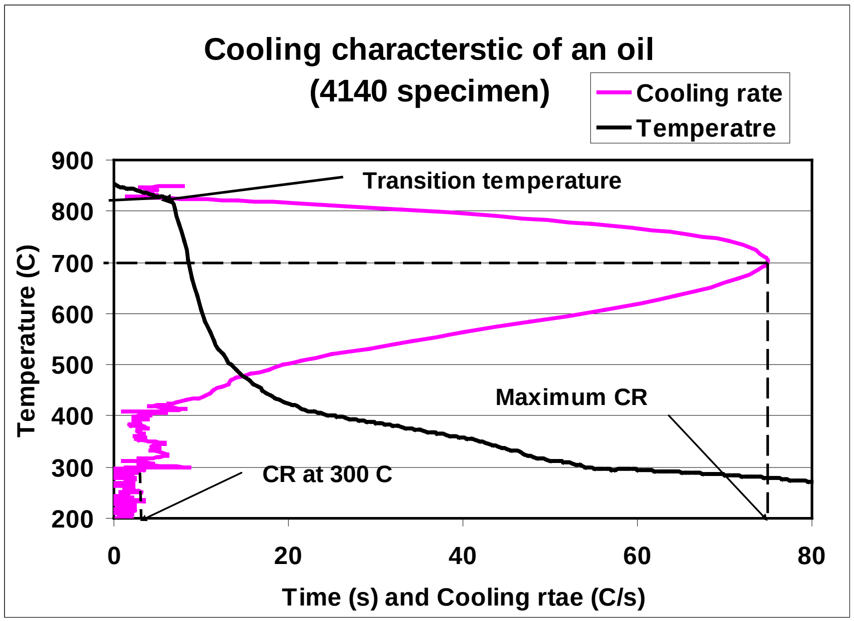
SEM at surface

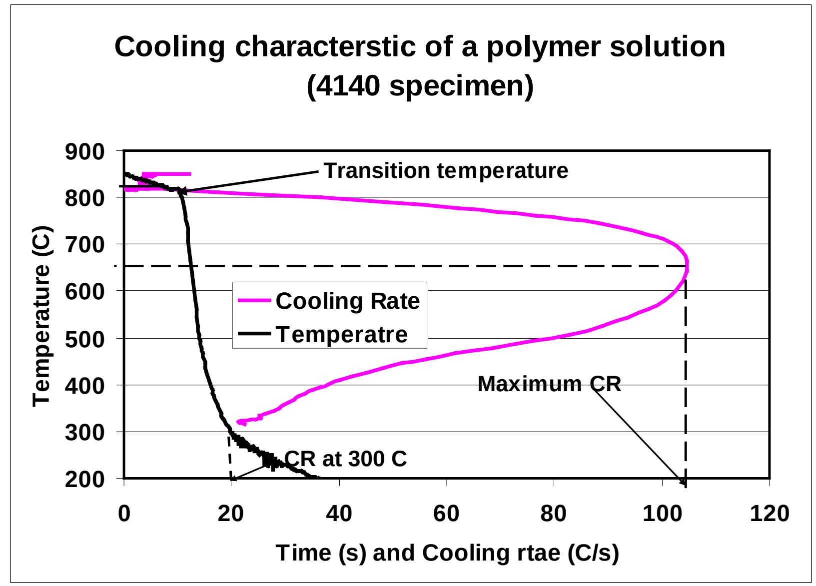


SEM at Core

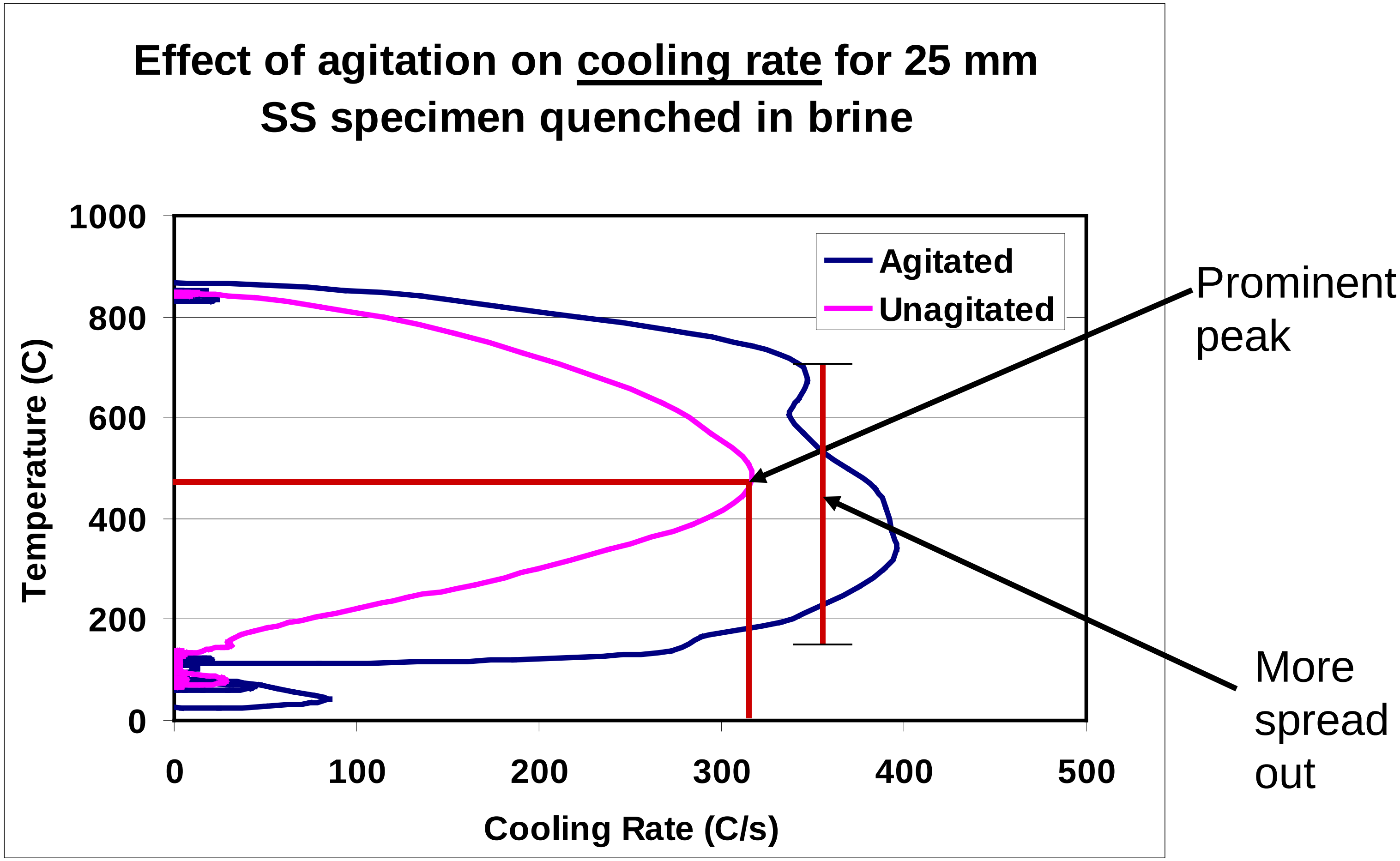


# “QuenchProbe”– Computation of Cooling Characteristics of Quenchants

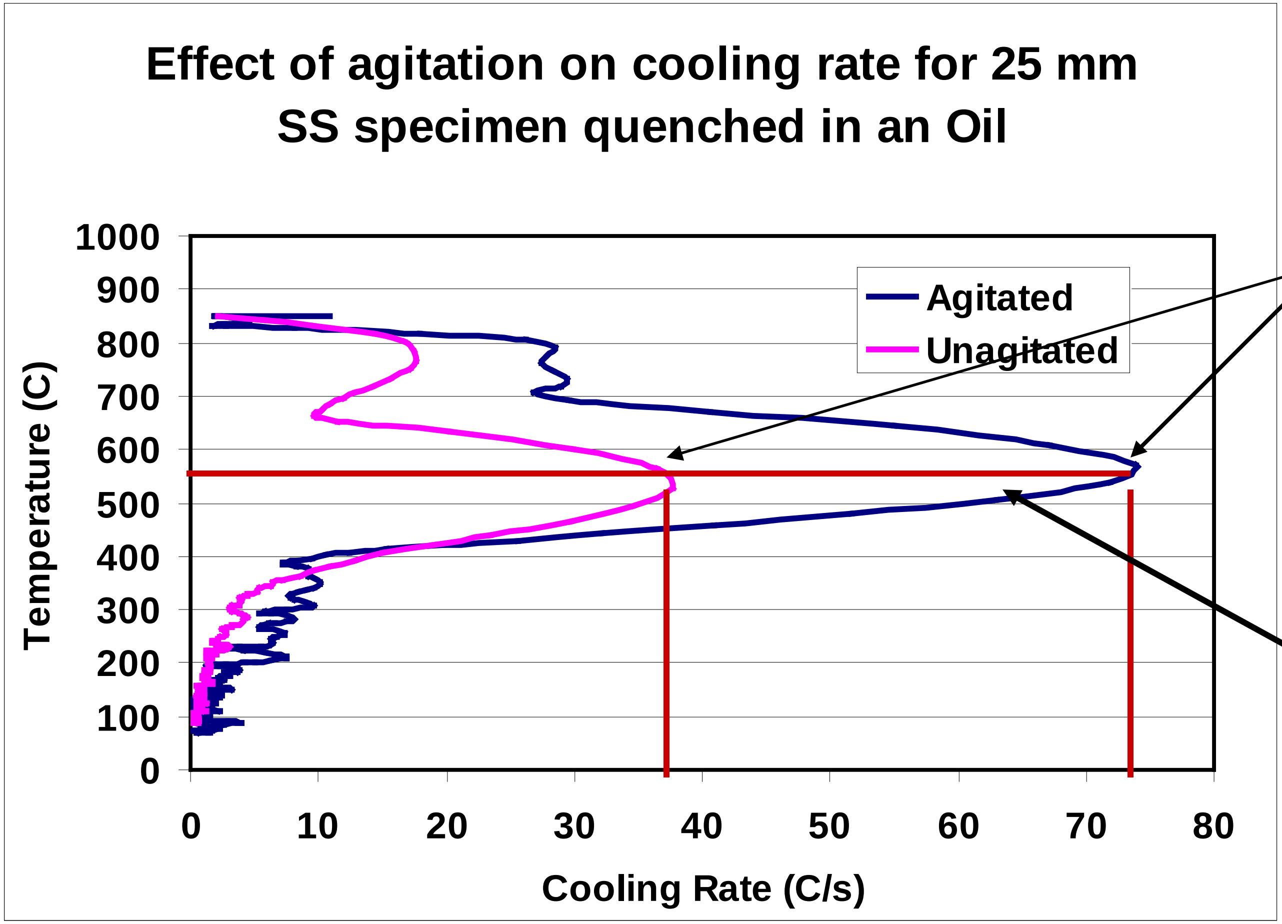


# "QuenchProbe" – Check Effect of Agitation on Cooling Rate (Brine)



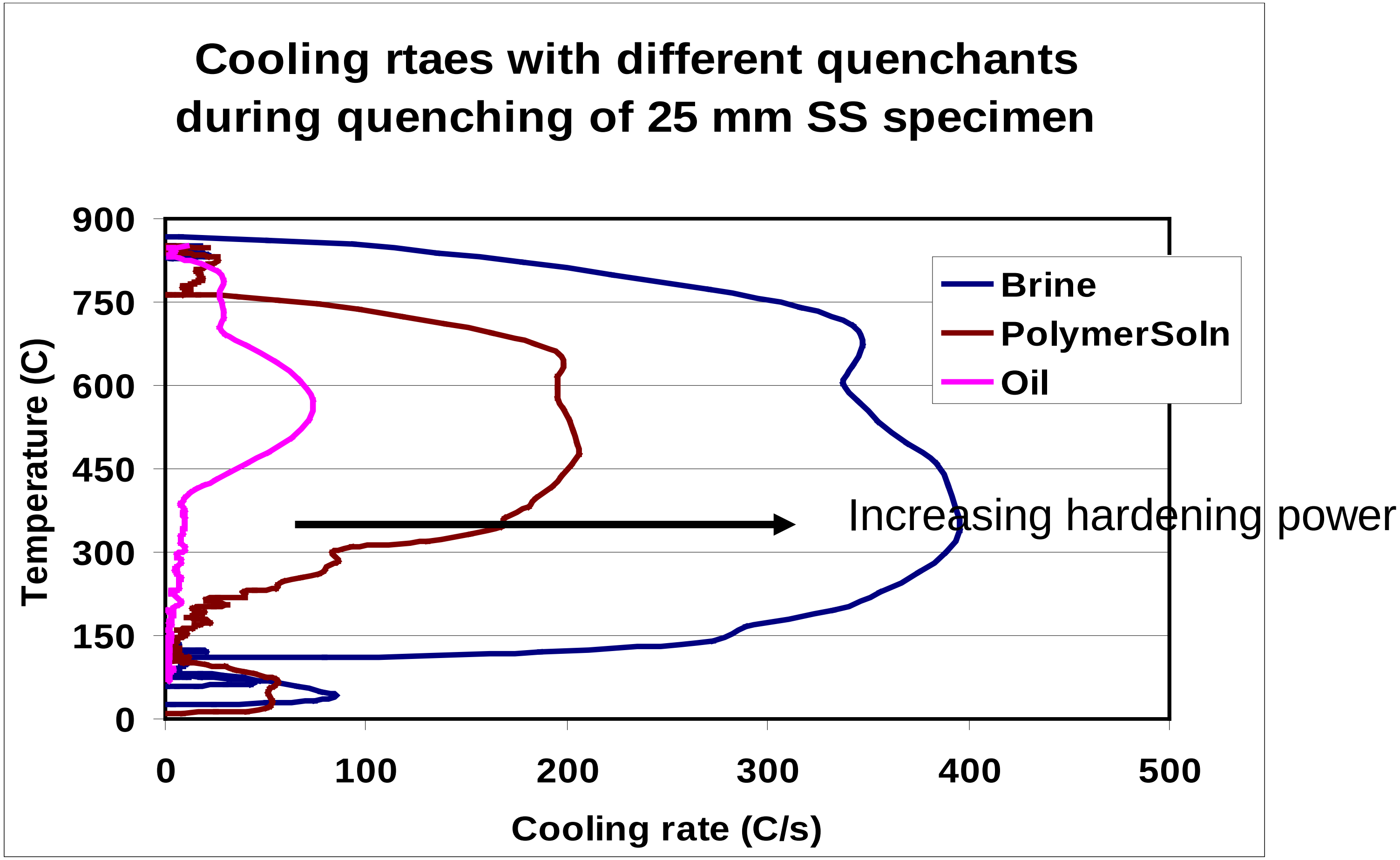
# "QuenchProbe" – Check Effect of Agitation on Cooling Rate (Oil)



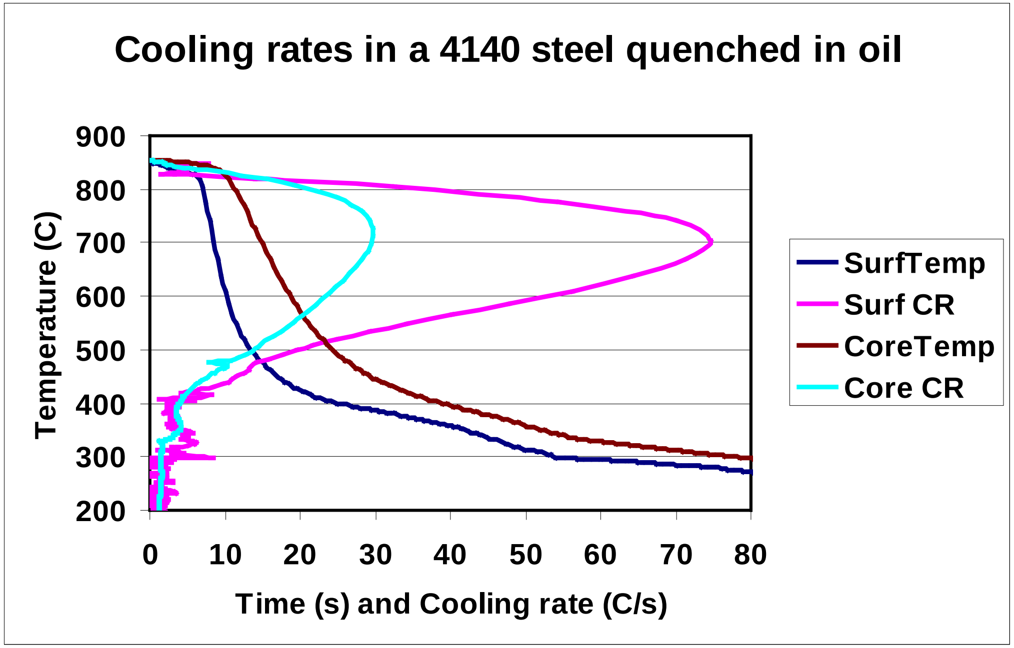

Doubled peak heat flux at the same temp

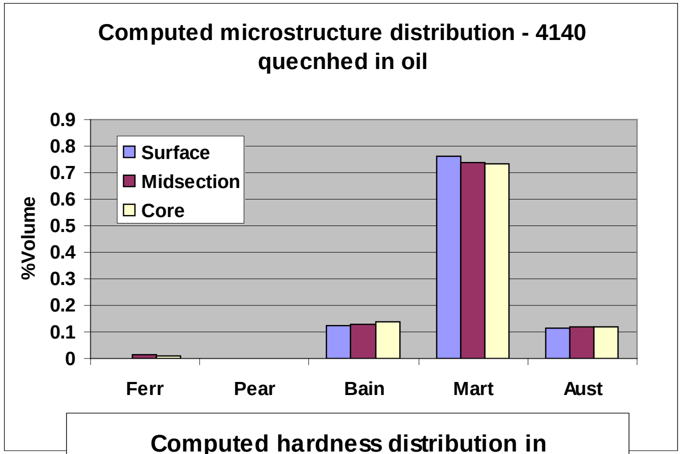
Higher heat flux due to agitation

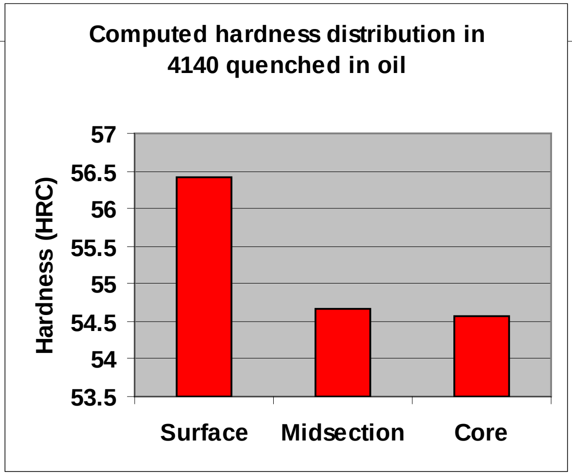
# "QuenchProbe"– Selection of Appropriate Quenchants



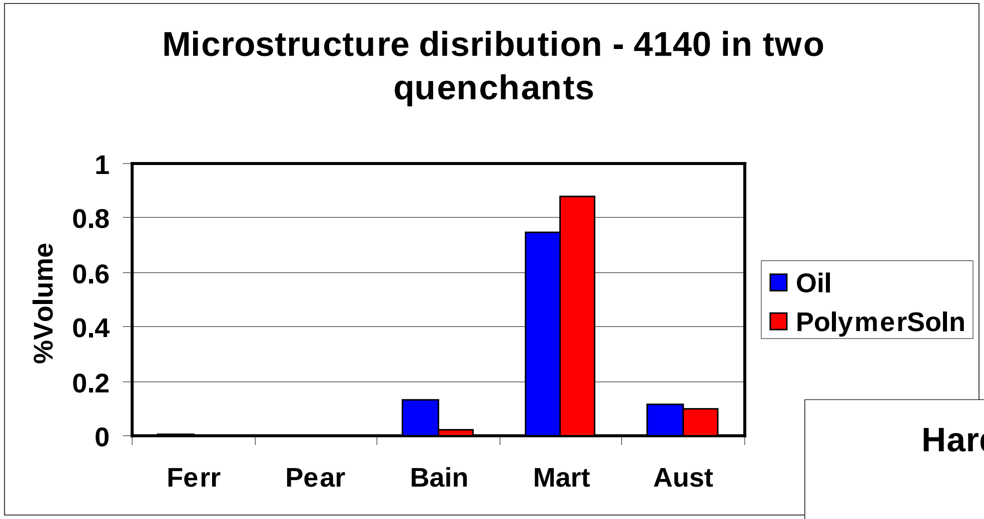
# “QuenchProbe”– Computation of Property Variations across Sections

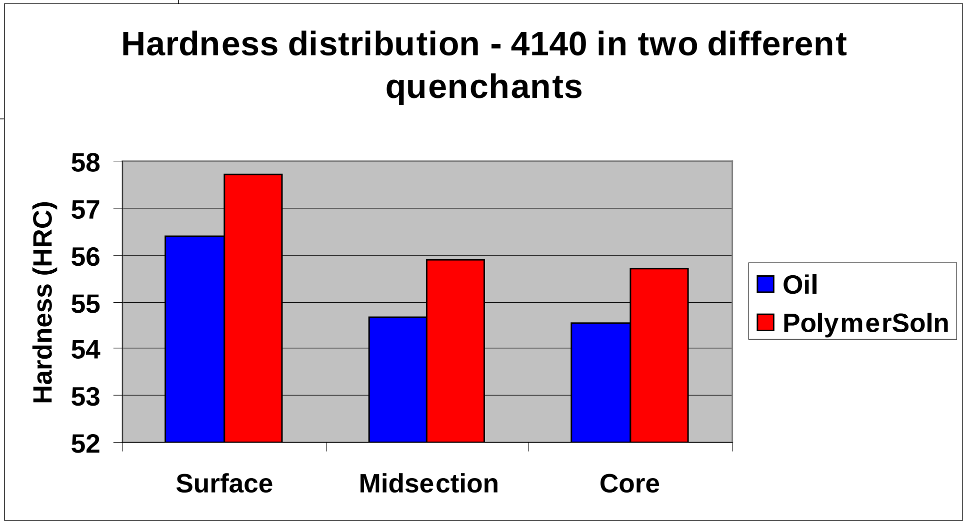


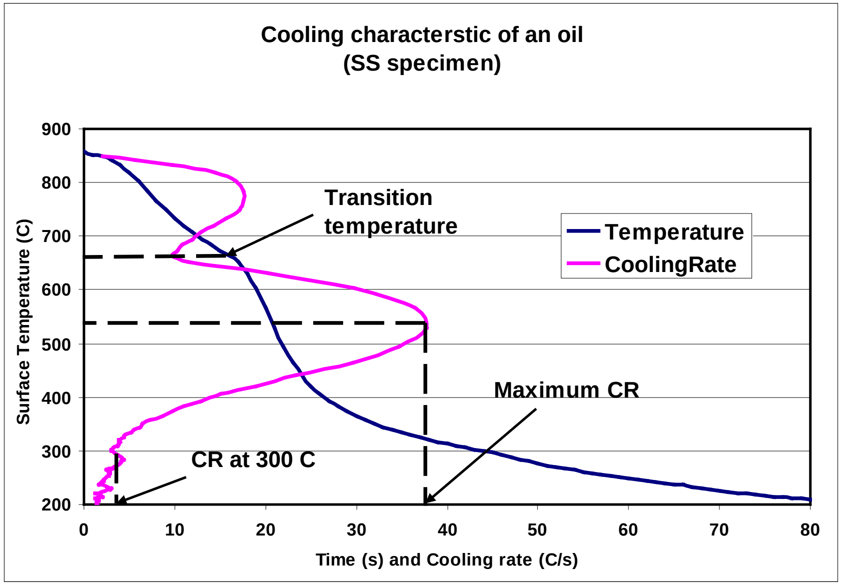



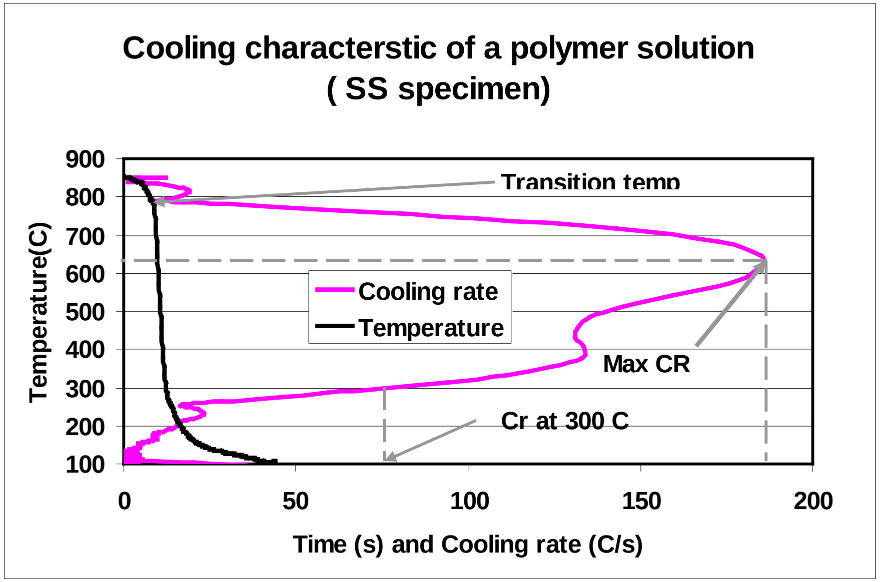
# "QuenchProbe"– Expected Microstructure and Hardness with Quenchants



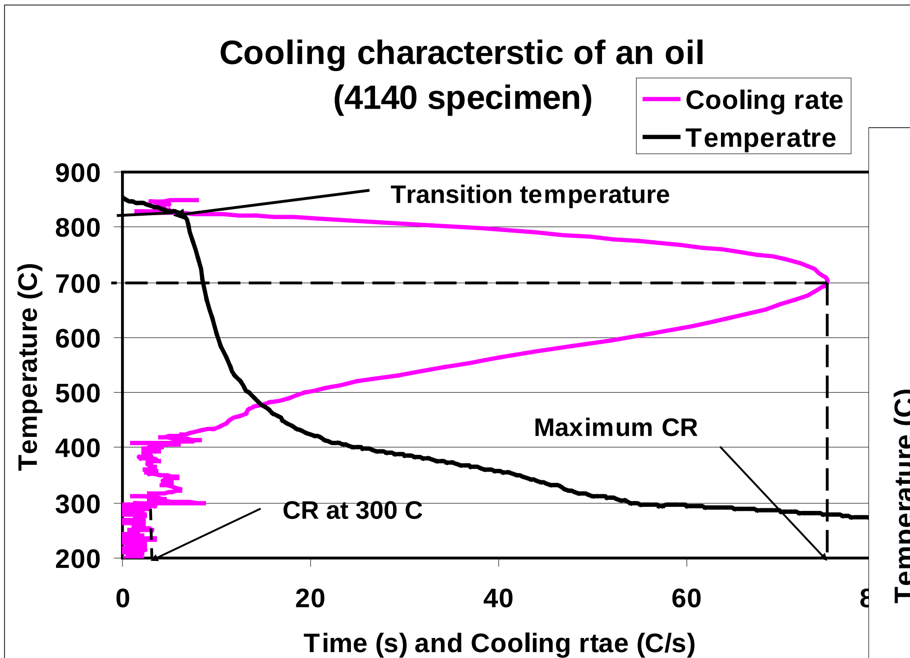



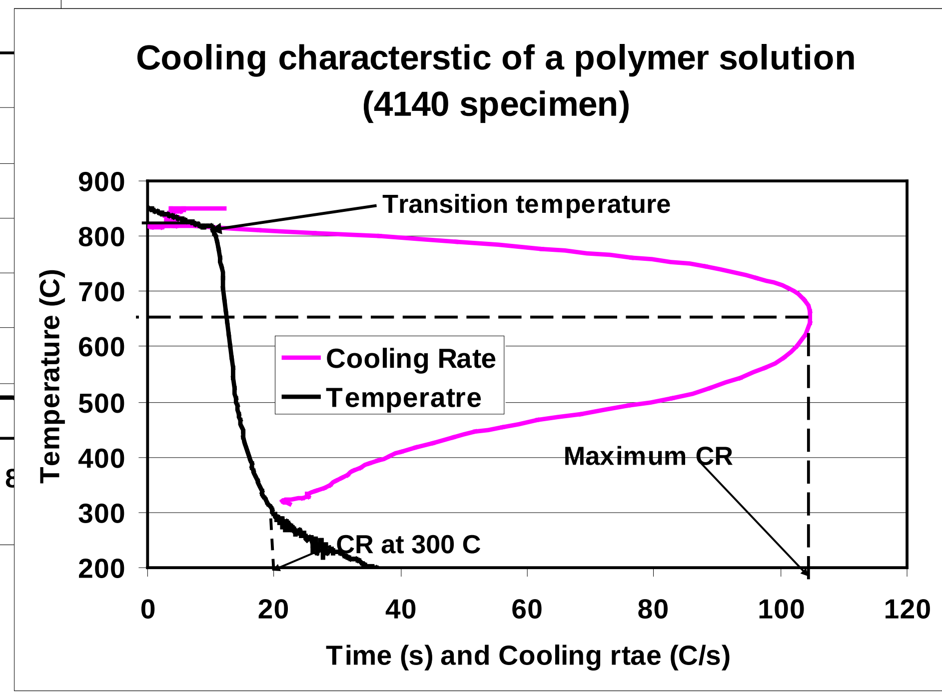
# "QuenchProbe" – Computation of Cooling Characteristics of Quenchants



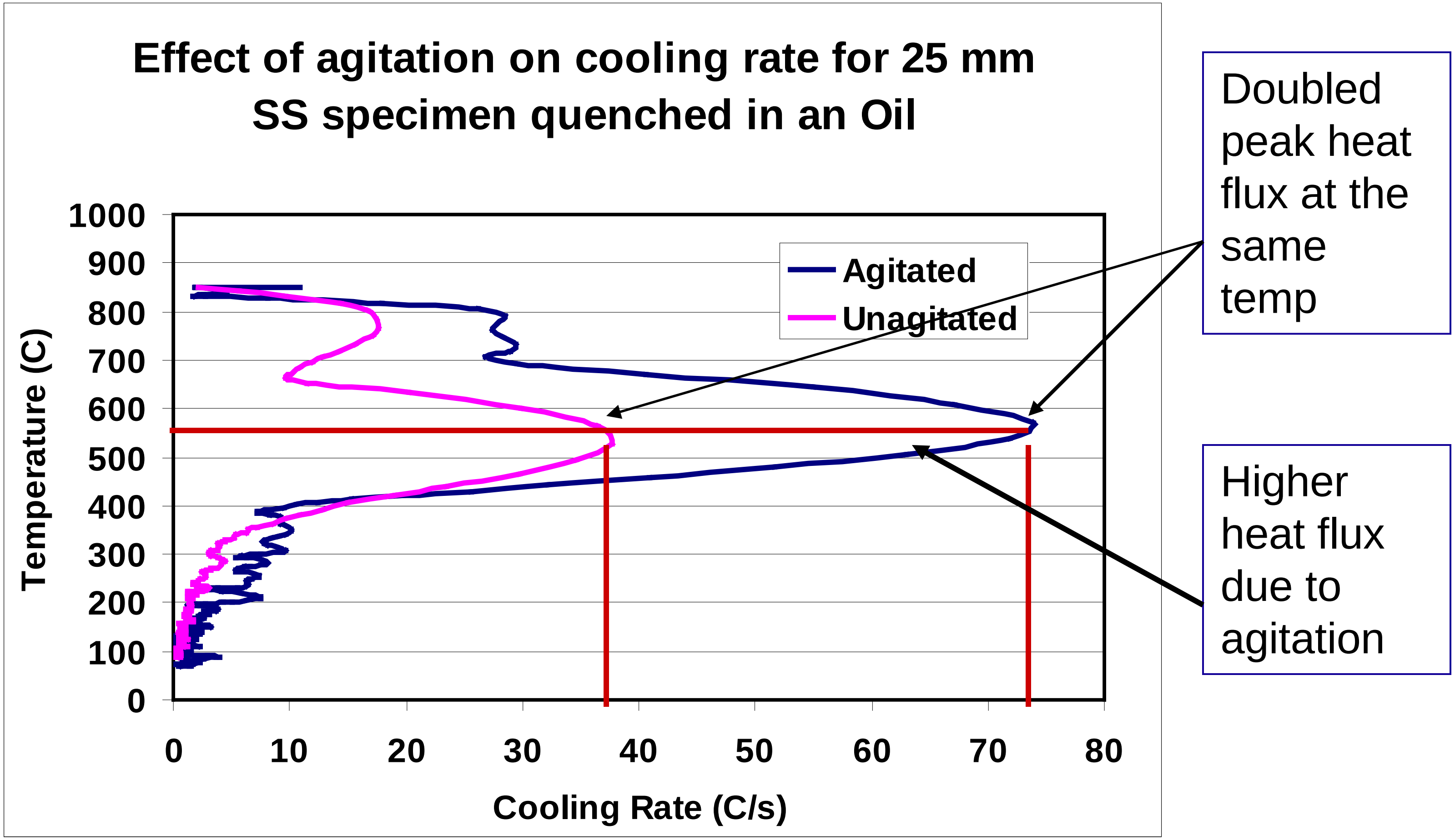



# "QuenchProbe"– Computation of Cooling Characteristics of Quenchants

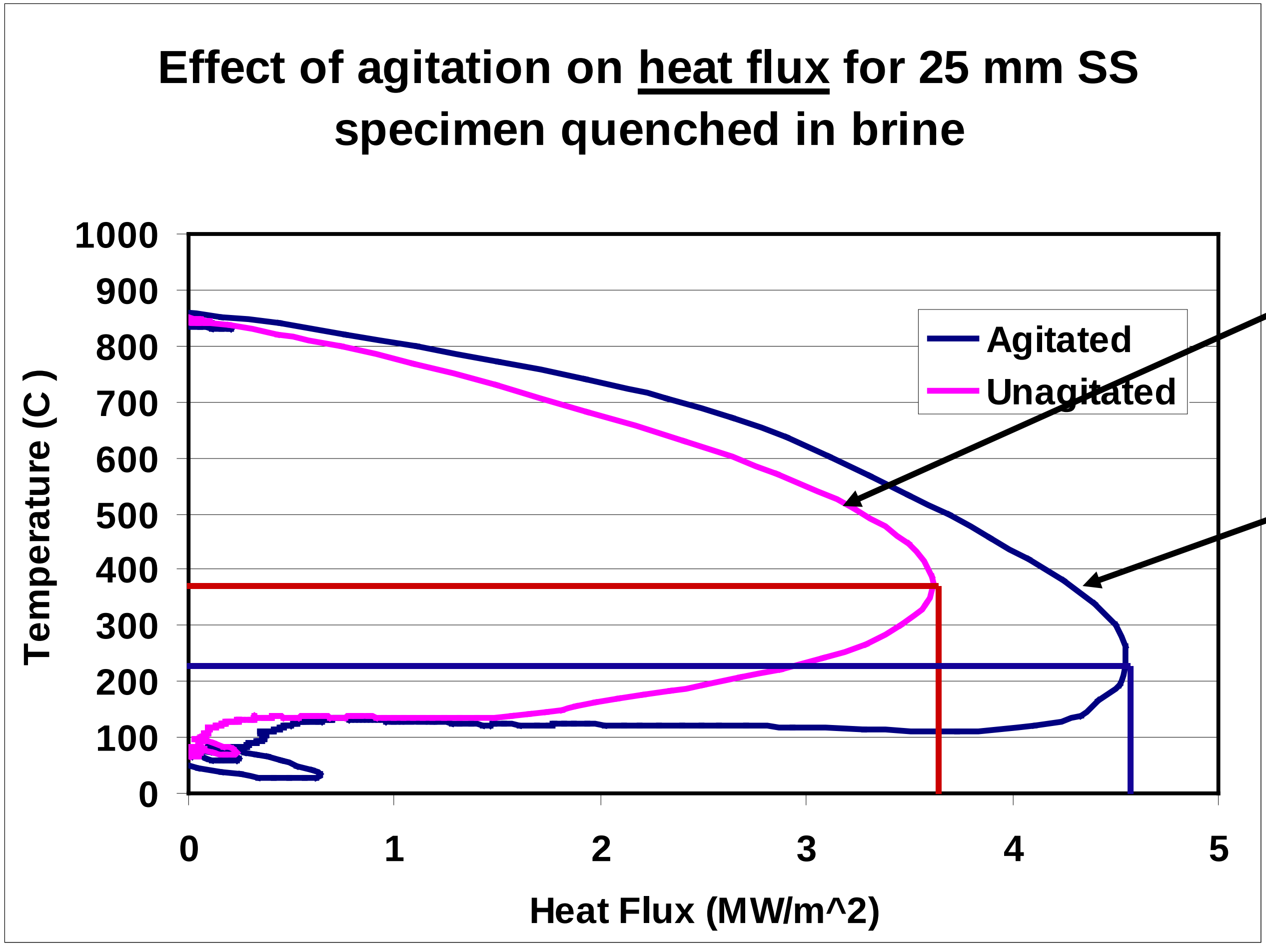


# "QuenchProbe" – Check Effect of Agitation on Cooling Rate (Oil)



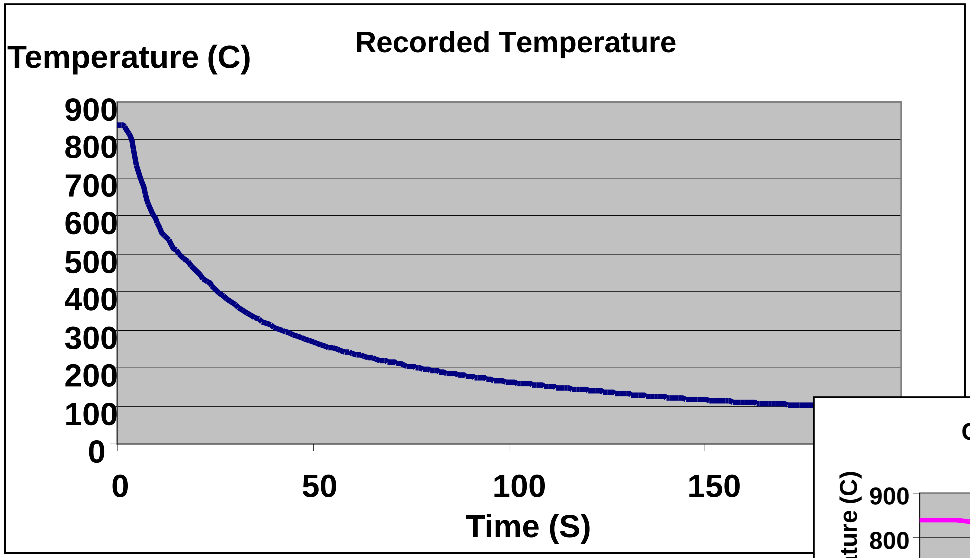
# "QuenchProbe" – Check Effect of Agitation on Heat Flux

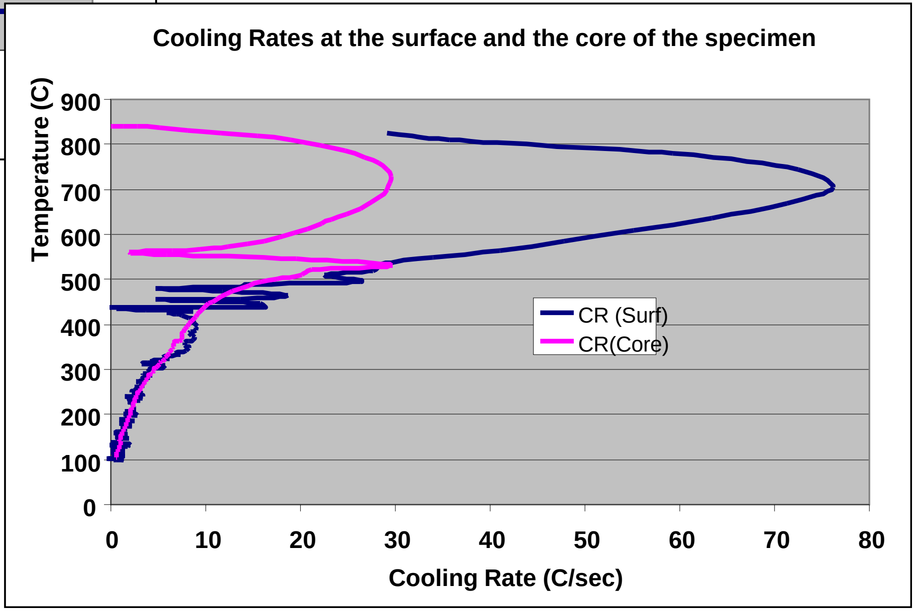



Lower heat flux; Peak at higher temp

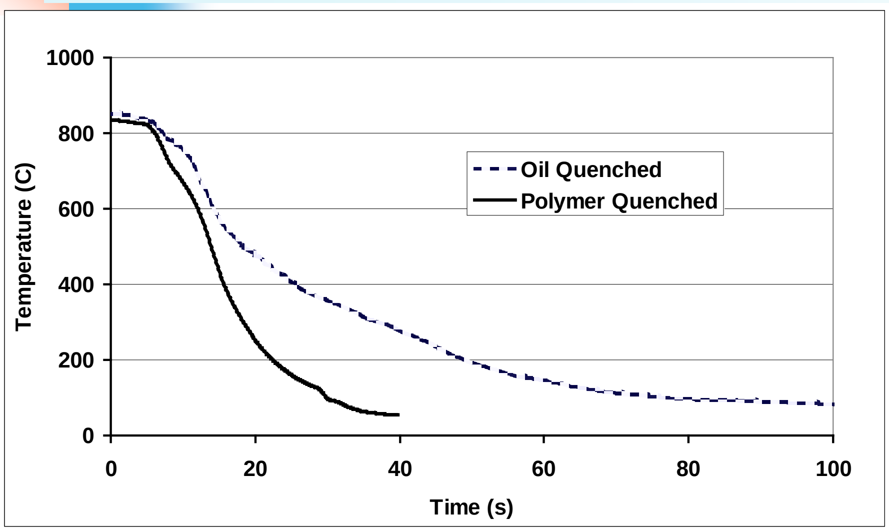
Higher heat flux; Peak at lower temp

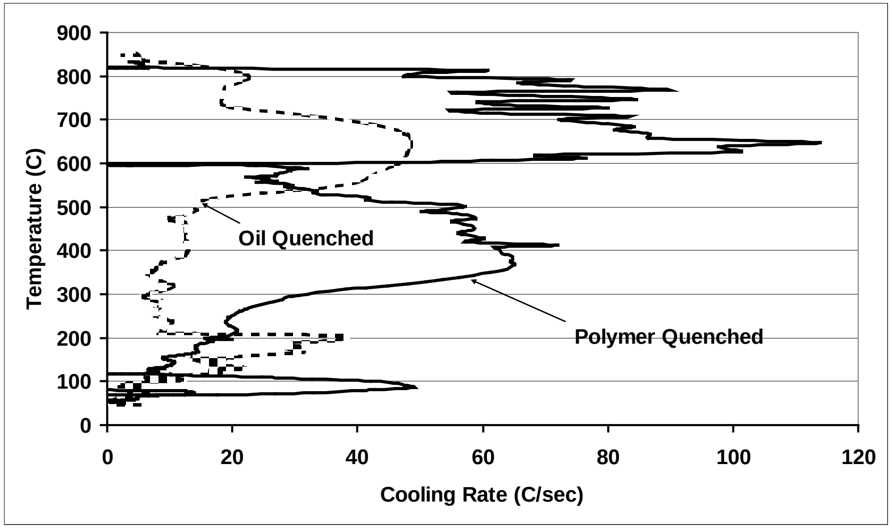
# Industrial Trials – 8822 H – Oil Quench

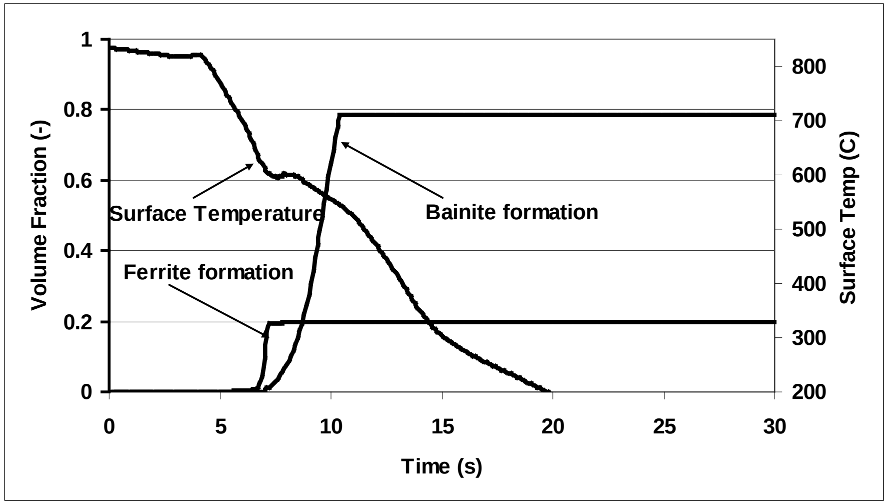


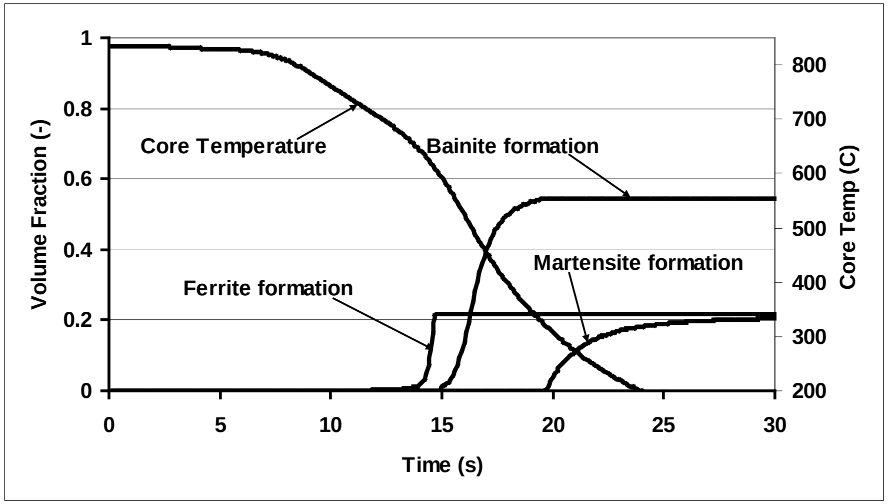



# Industrial Trials – C 45 – Oil and Polymer Quenched

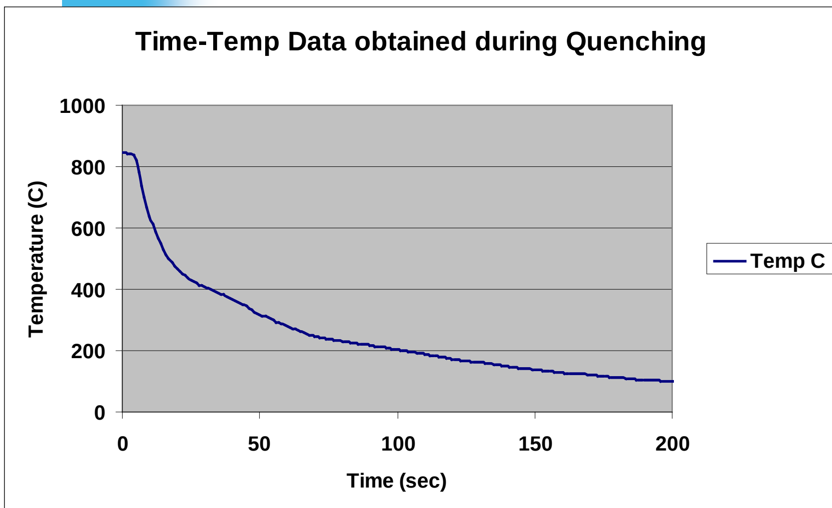


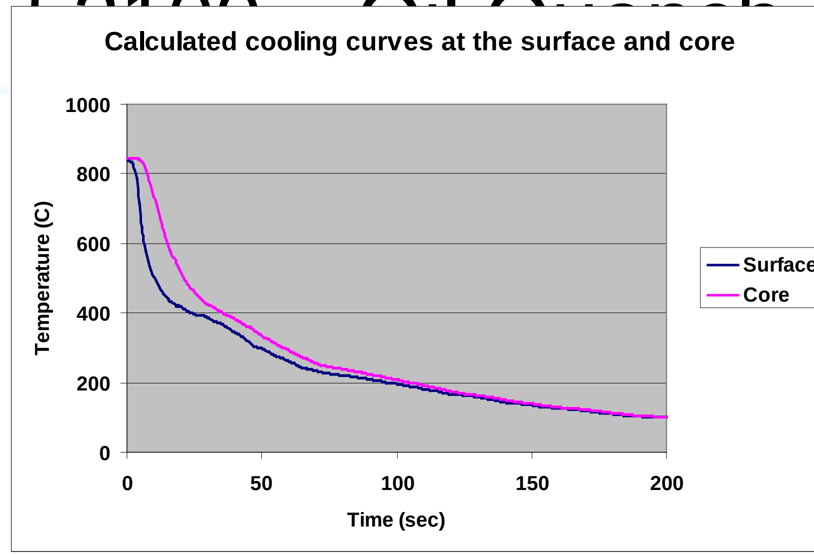



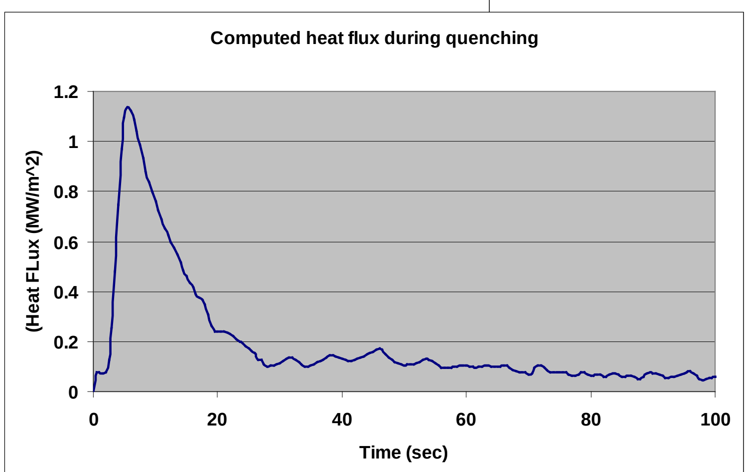



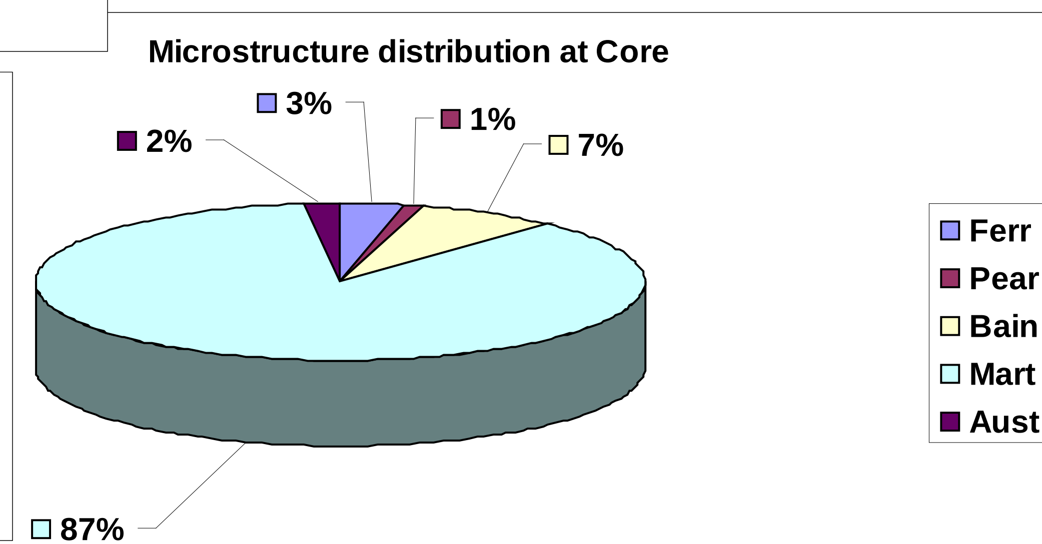



# Industrial Trials – SAE

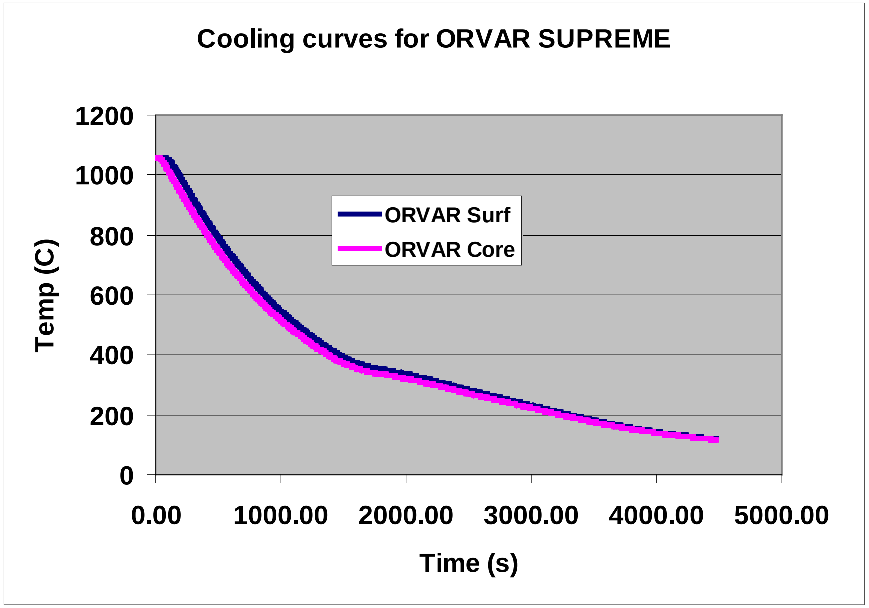


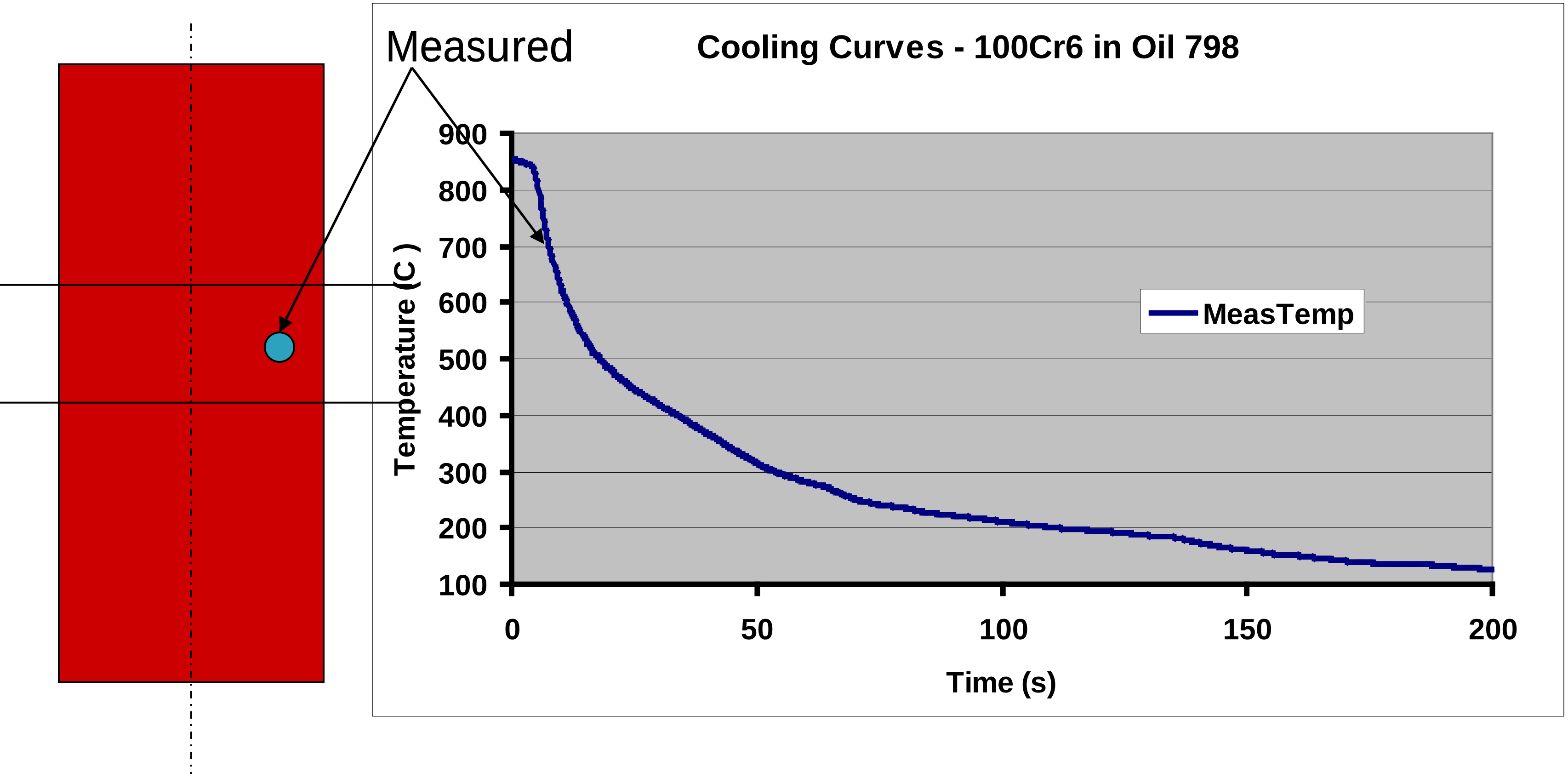
# Industrial Trials – Vacuum Quench



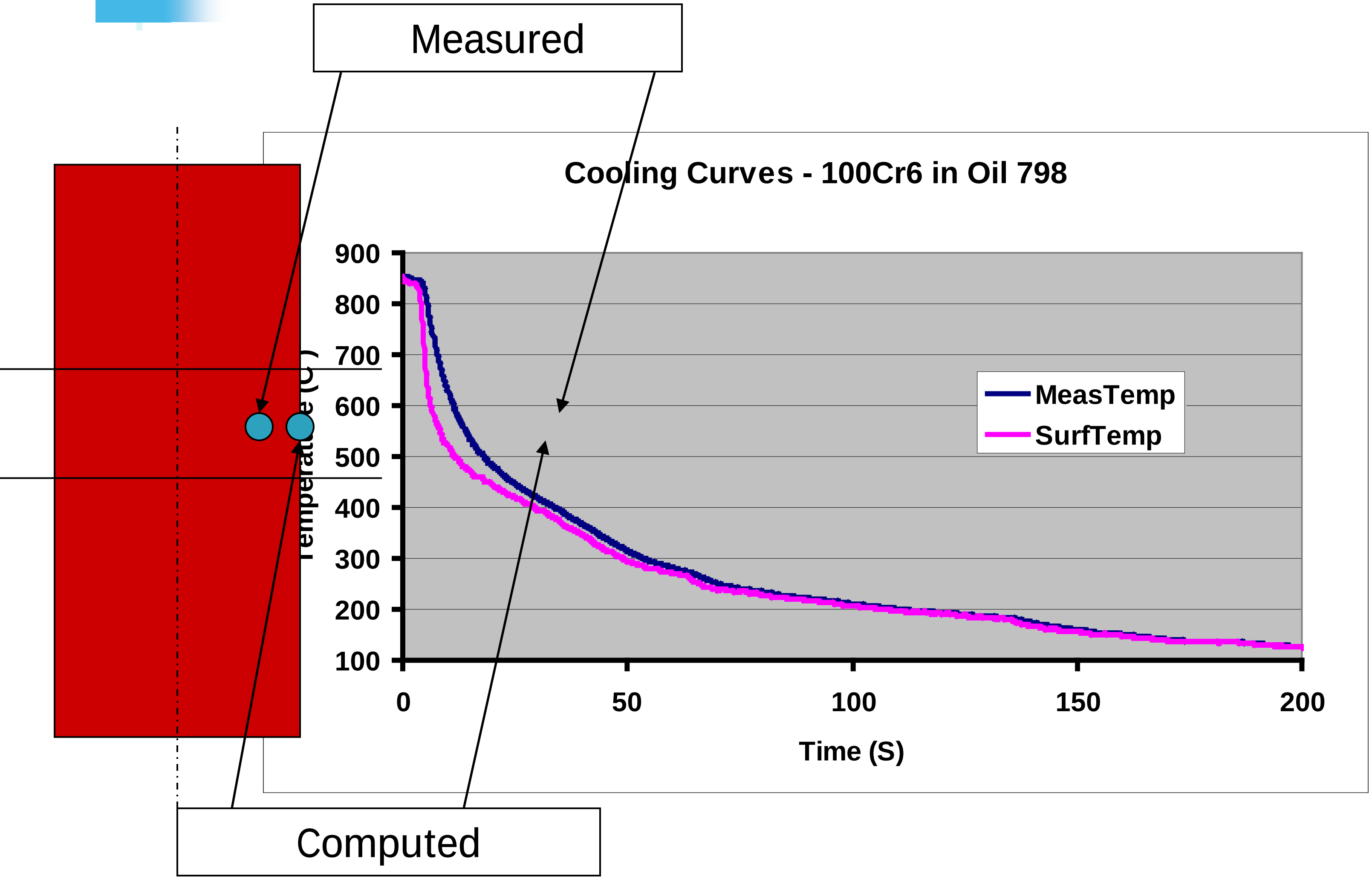

| Sl No | Sample ID | HRC | | Error % |
|---|---|---|---|---|
| | | Measured | Computed | |
| 1 | DAC Surface | 55.0 | 54.51 | -0.89 |
| 2 | DAC Core | 54.7 | 54.50 | -0.37 |
| 3 | H13 Surface | 55.0 | 54.49 | -0.93 |
| 4 | H13 Core | 55.4 | 54.50 | -1.65 |
| 5 | ORVAR Surface | 55.0 | 54.59 | -0.75 |
| 6 | ORVAR Core | 55.7 | 54.59 | -2.03 |

**Comparison of measured and computed values of hardness**

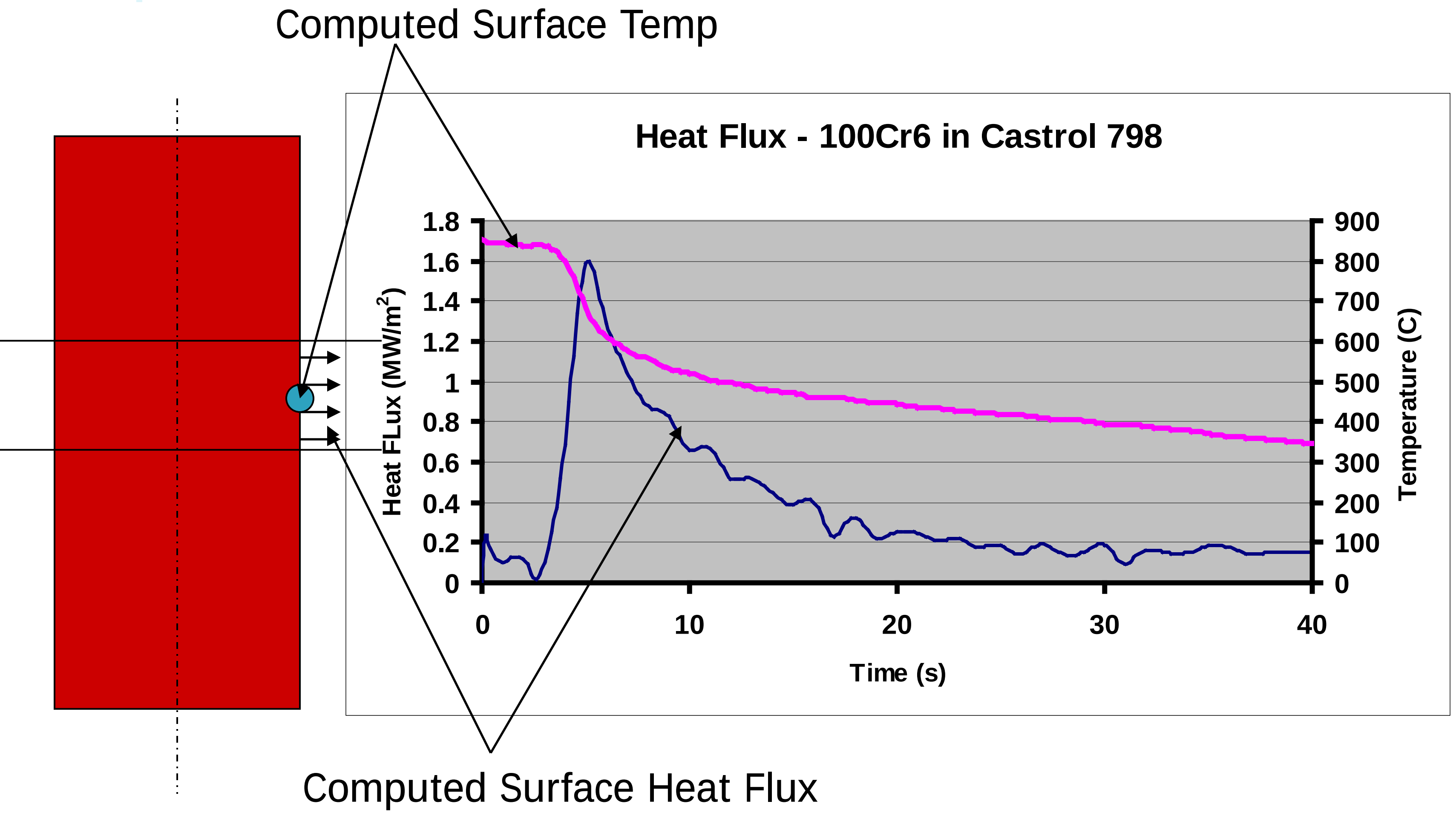
# Plant test - 100Cr6 in Castrol 798



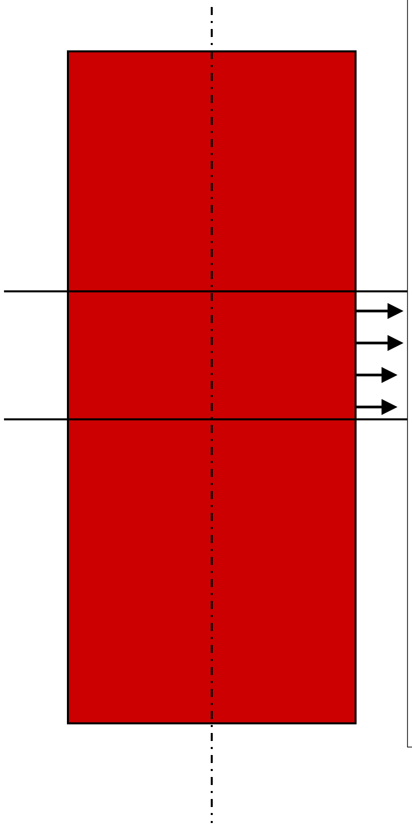
# HTC Determination – 100Cr6 in Castrol 798

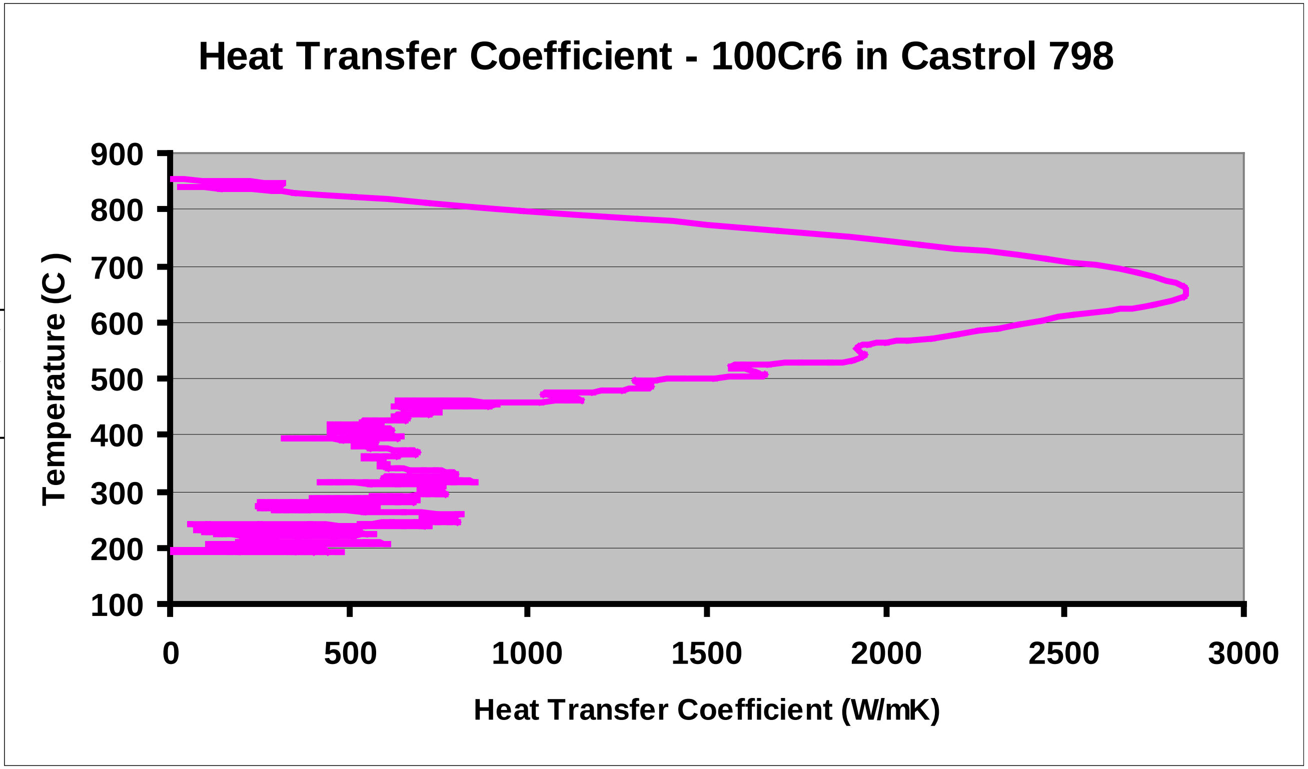


# HTC Determination – 100Cr6 in Castrol 798

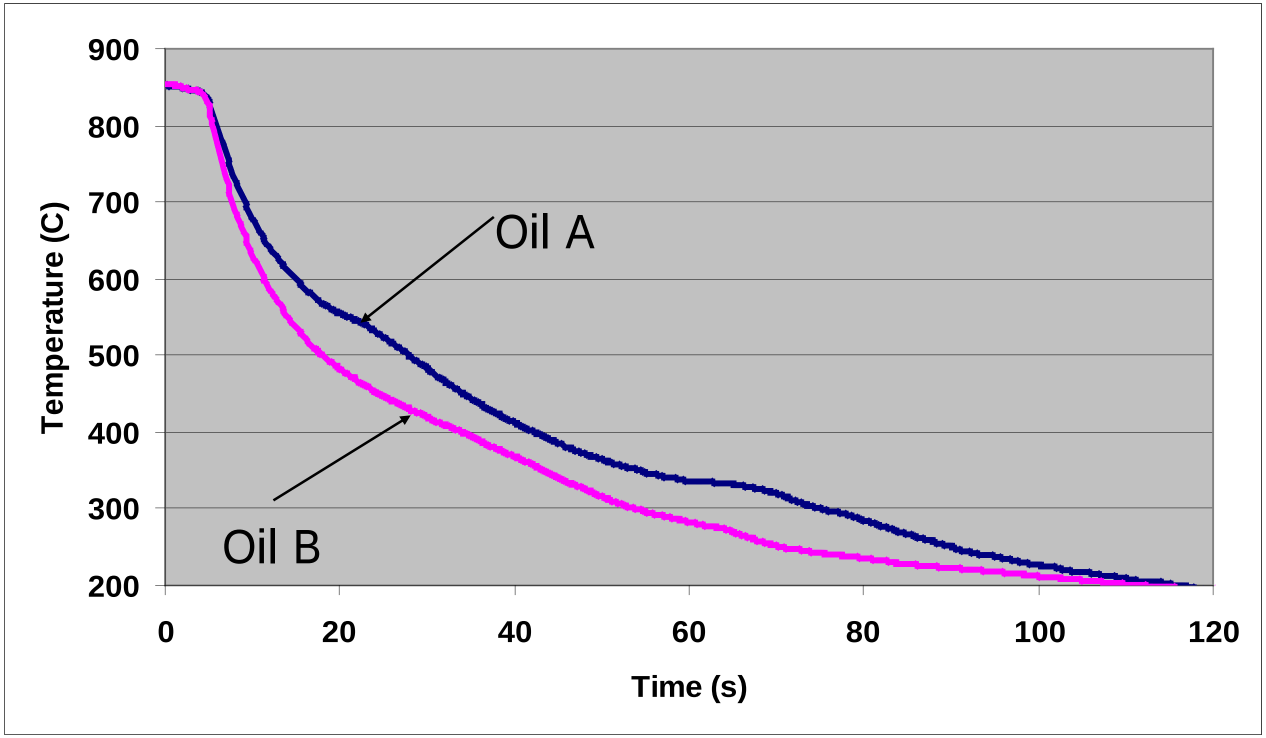


# HTC Determination – 100Cr6 in Castrol 798





# Performance of different Oils in Plant Conditions

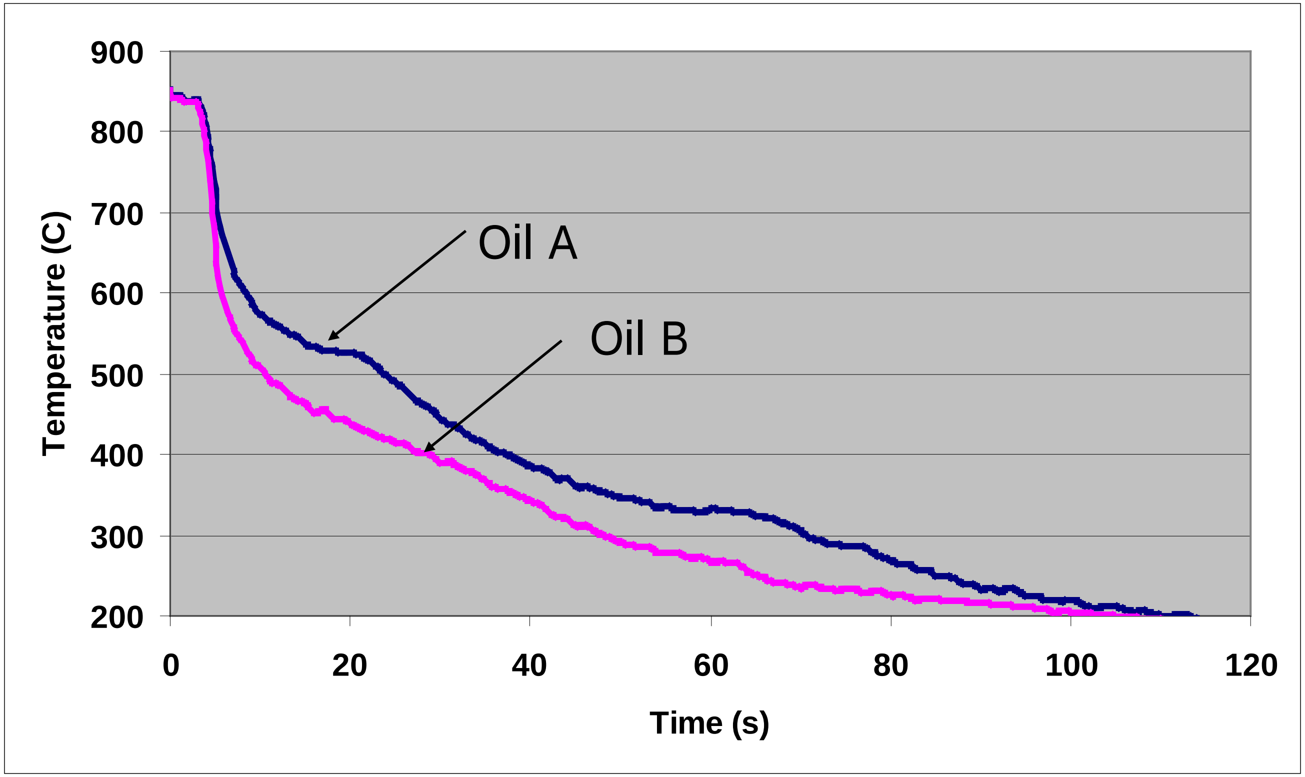



Cooing curves obtained with **100Cr6** steel :

C: 1.04%; Mn:0.33%; Si:0.26%; Cr:1.53%; Cu:0.20%; Ni:0.31%; P:0.023%; S:0.006%

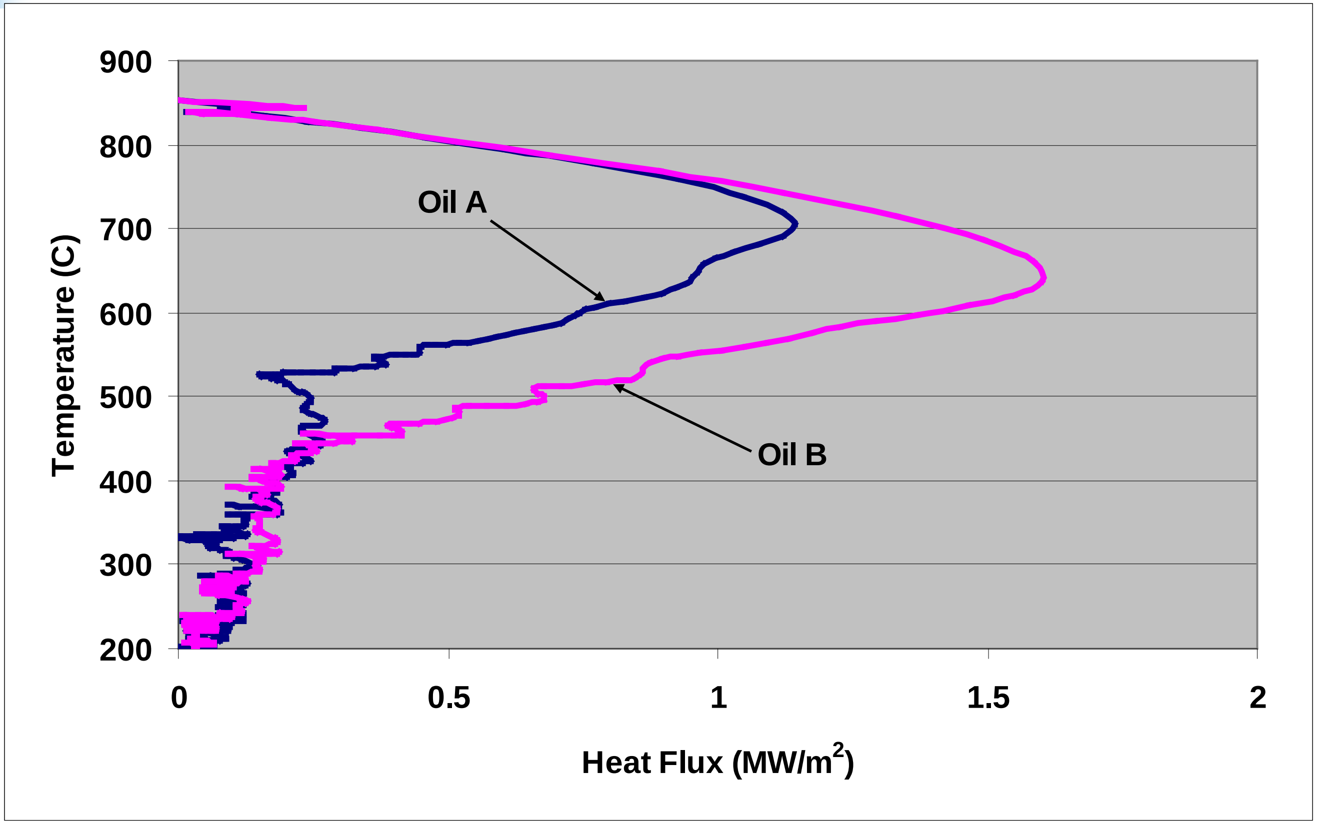
**Oil A**: Nippon Hot Quench Oil 303; **Oil B**: Castrol Iloquench 798 Hot Quenching Oil

# Performance of different Oils in Plant Conditions



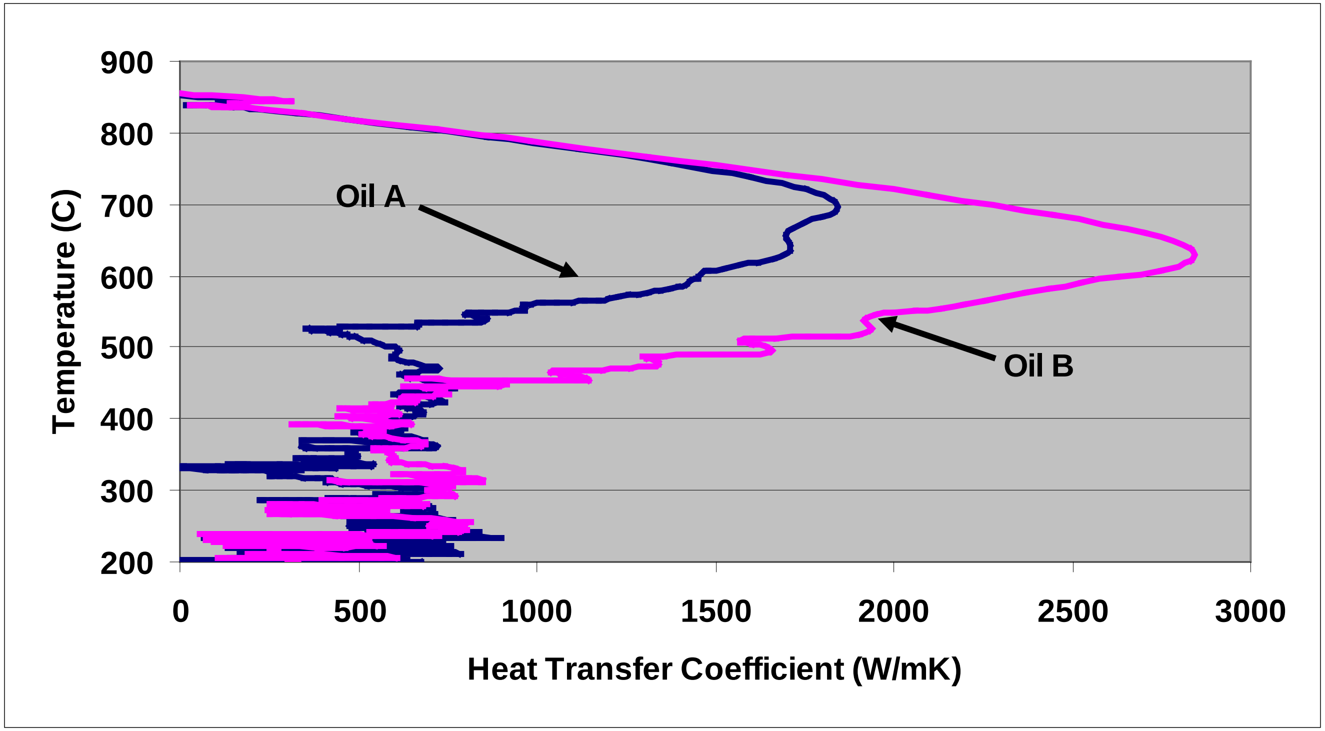

**Comparison of cooling curves at the surface of the quenched specimen computed using the measured temperature data**

# Performance of different Oils in Plant Conditions



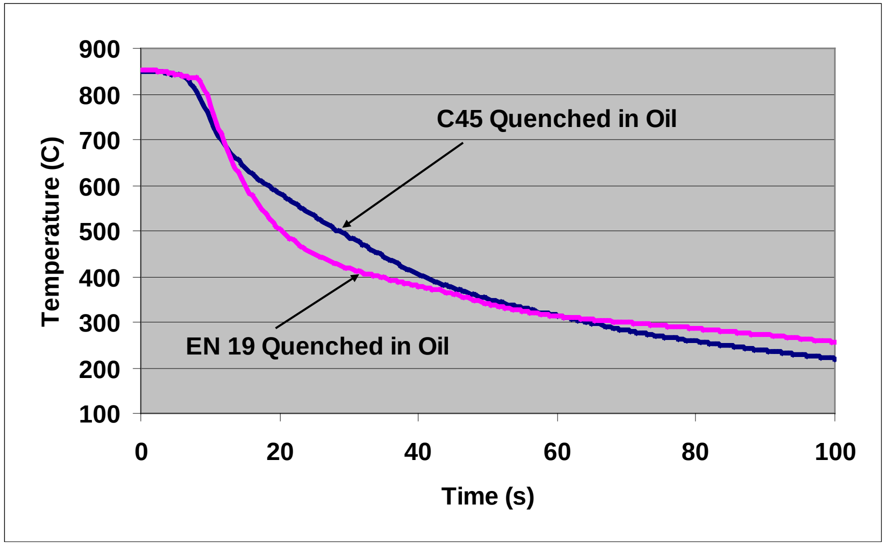

**Surface heat flux during quenching of a 25 mm diameter 100Cr6 grade steel shown as a function of surface temperature, for the two oils.**

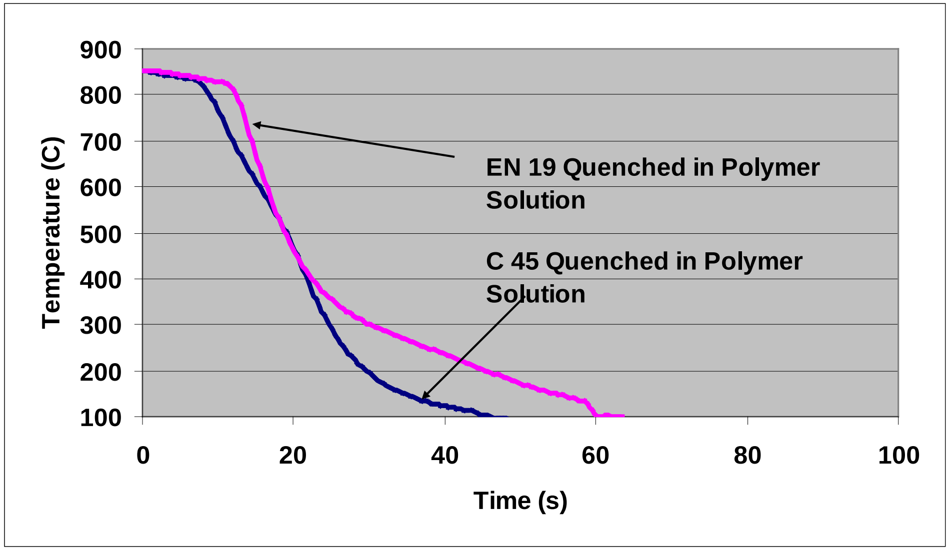
# Performance of different Oils in Plant Conditions




**Comparison of heat transfer coefficient as function of surface temperature during immersion quenching of a 25 mm diameter 100Cr6 grade steel in two Oils.**

# Cooling Curves for Alloy Steels

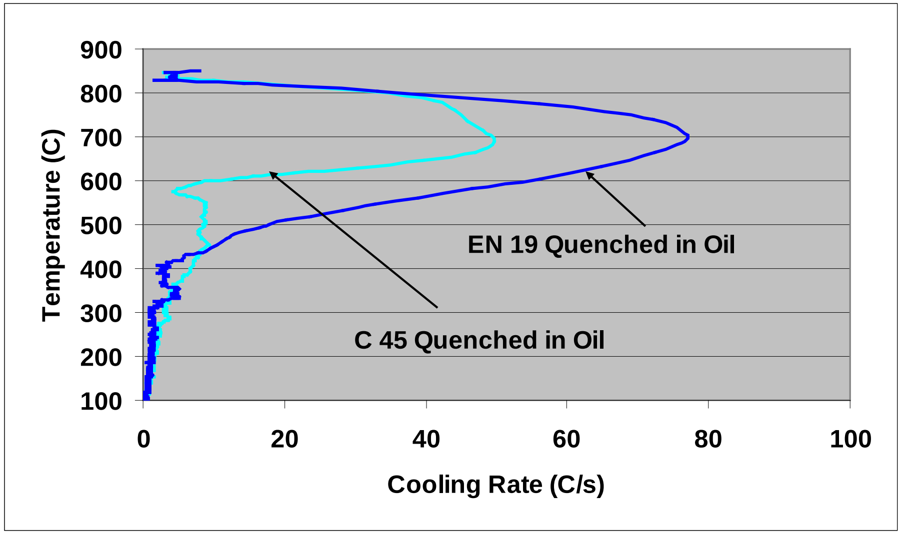


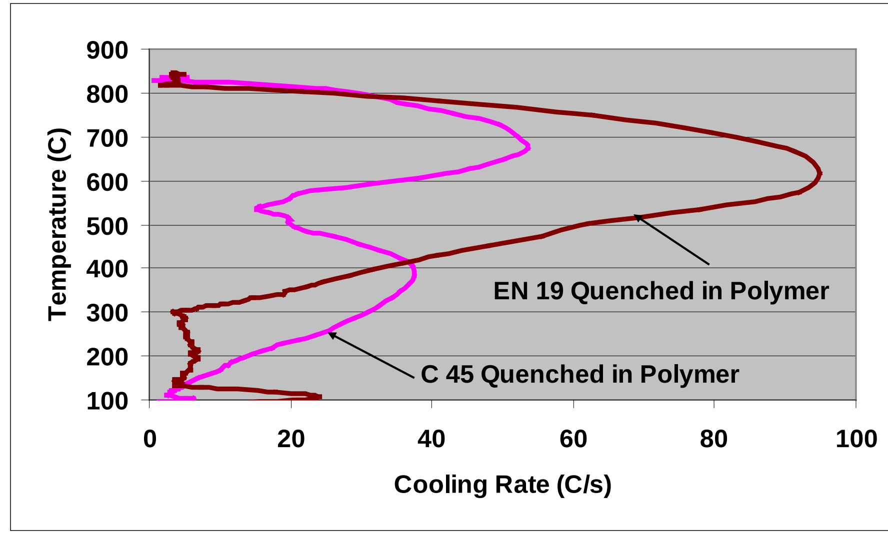




Recorded cooling curves obtained during quenching of **C 45** and **EN 19** steel specimens in a **mineral oil**

Recorded cooling curves obtained during quenching of **C 45** and **EN 19** steel specimens in an **aqueous solution of polymer**

# Cooling Rate Curves for Alloy Steels

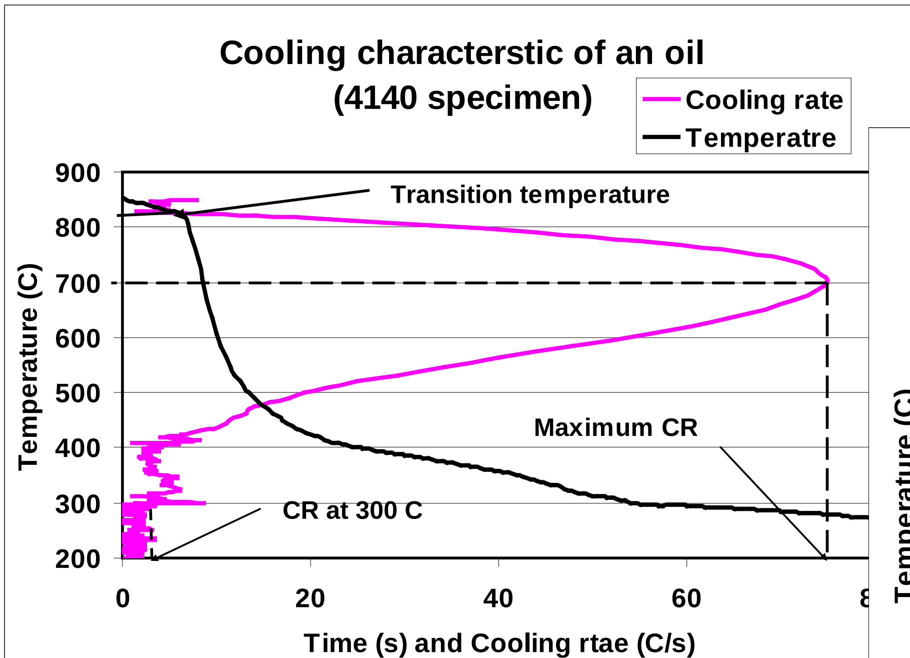


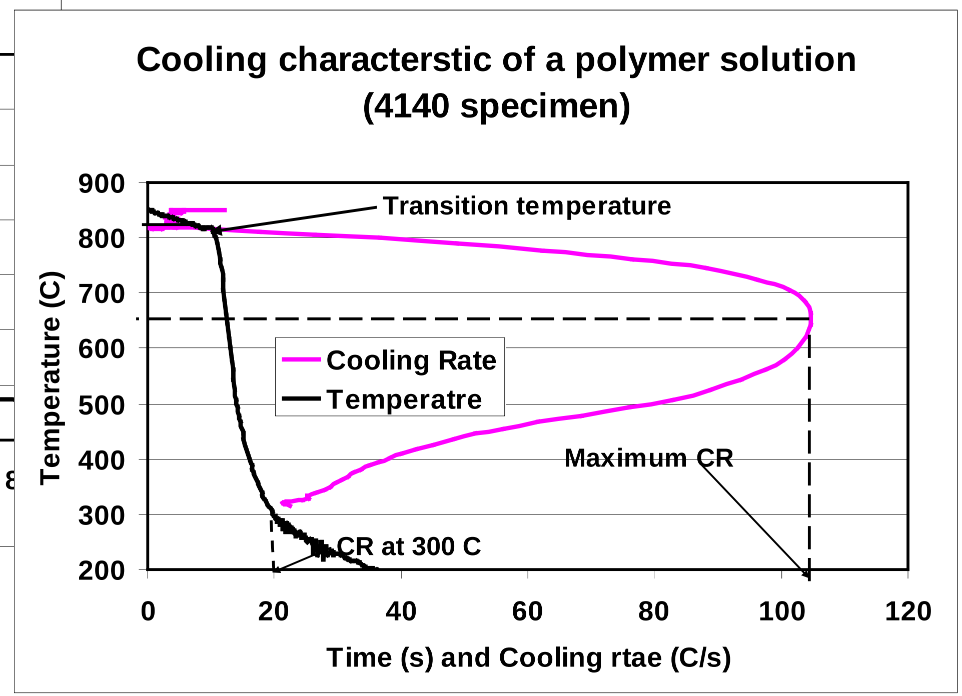




Computed surface cooling rates for steel specimens of **different grades** quenched in identical **mineral oils**

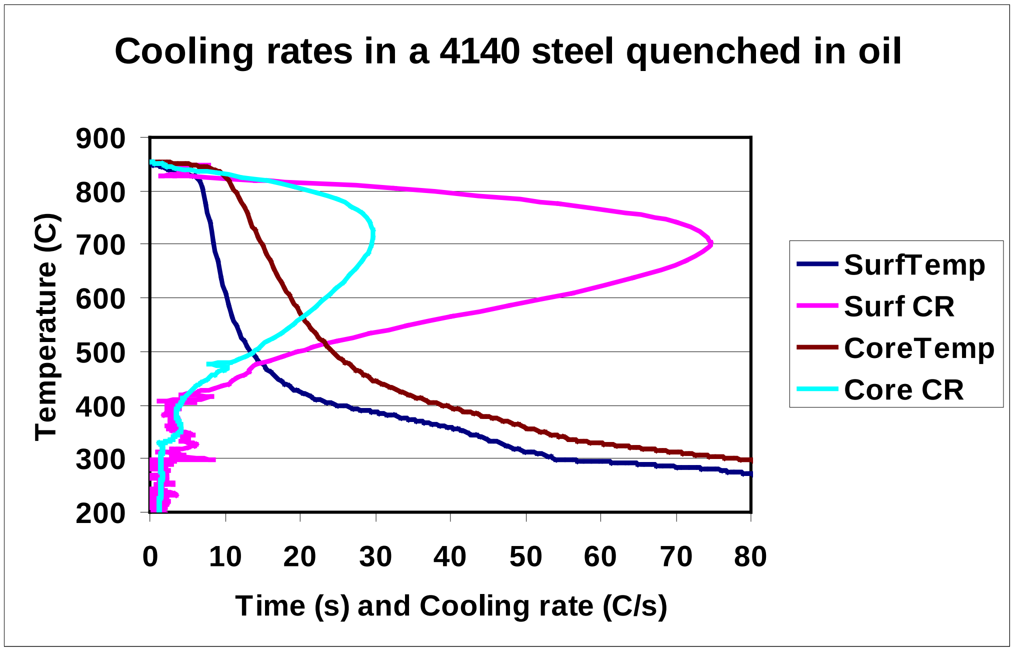
Computed surface cooling rates for steel specimens of **different grades** quenched in identical aqueous **solutions of polymer**

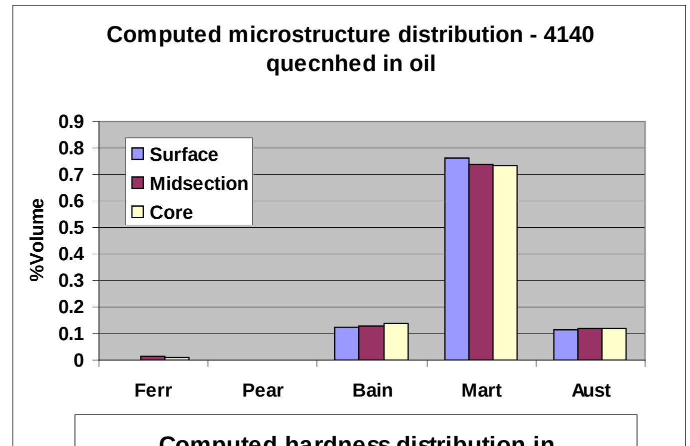
# "QuenchProbe"– Computation of Cooling Characteristics of Quenchants

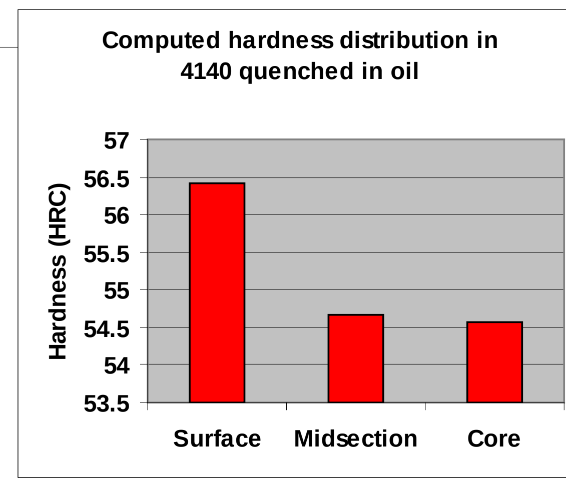


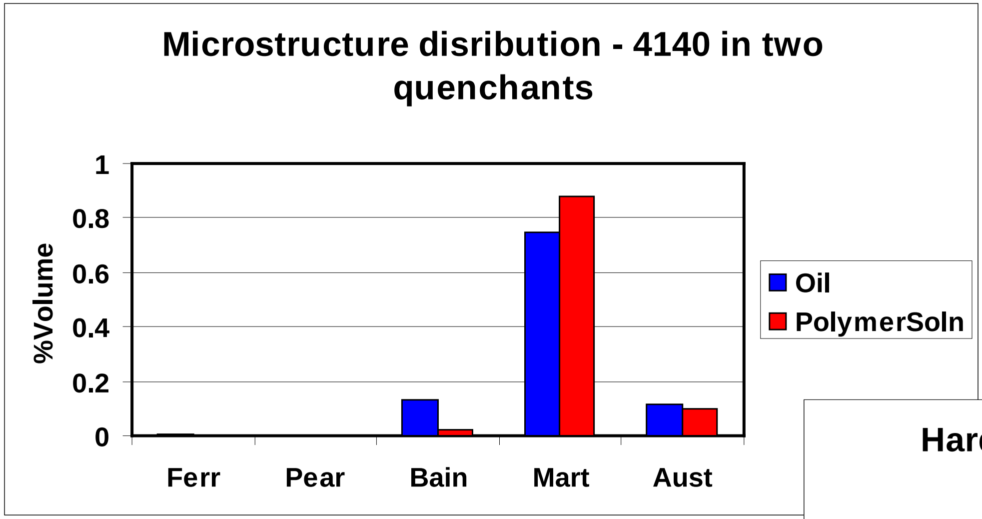
# "QuenchProbe"– Computation of Property Variations across Sections

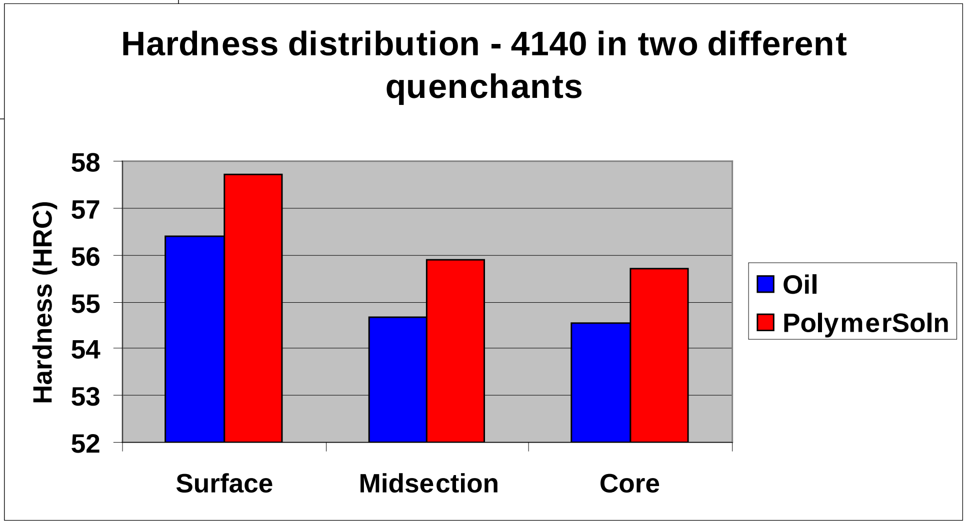


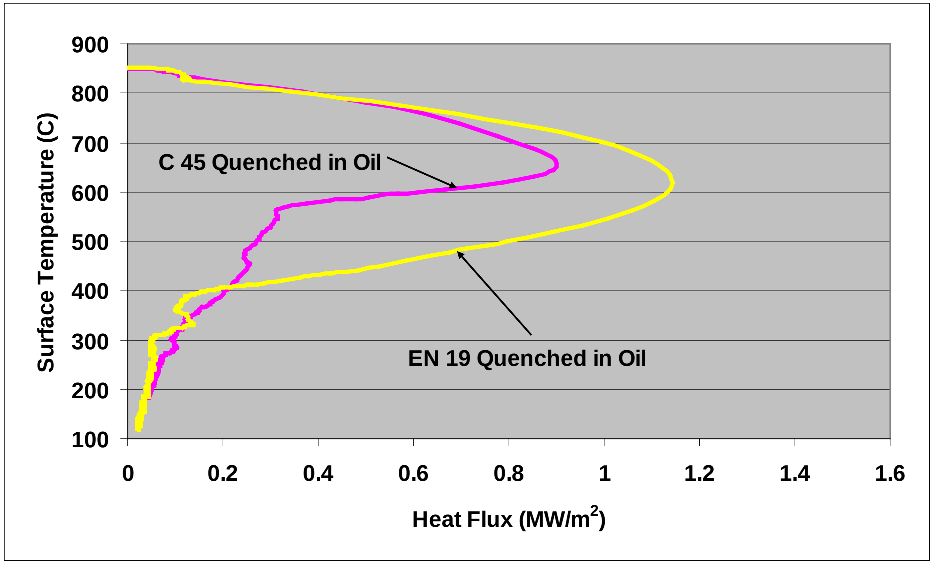



# “QuenchProbe”– Expected Microstructure and Hardness with Quenchants

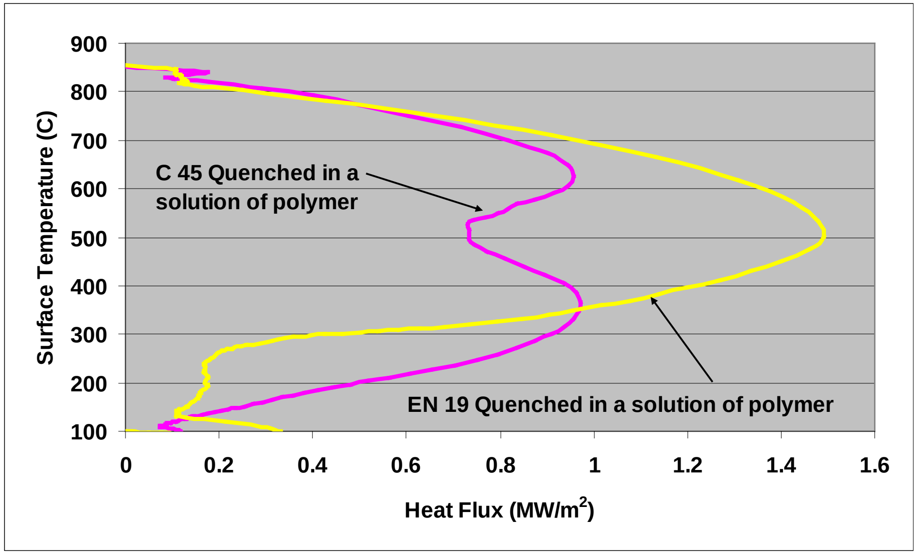


# Cooling Curve Analysis for Alloy Steels by "QuenchProbe"

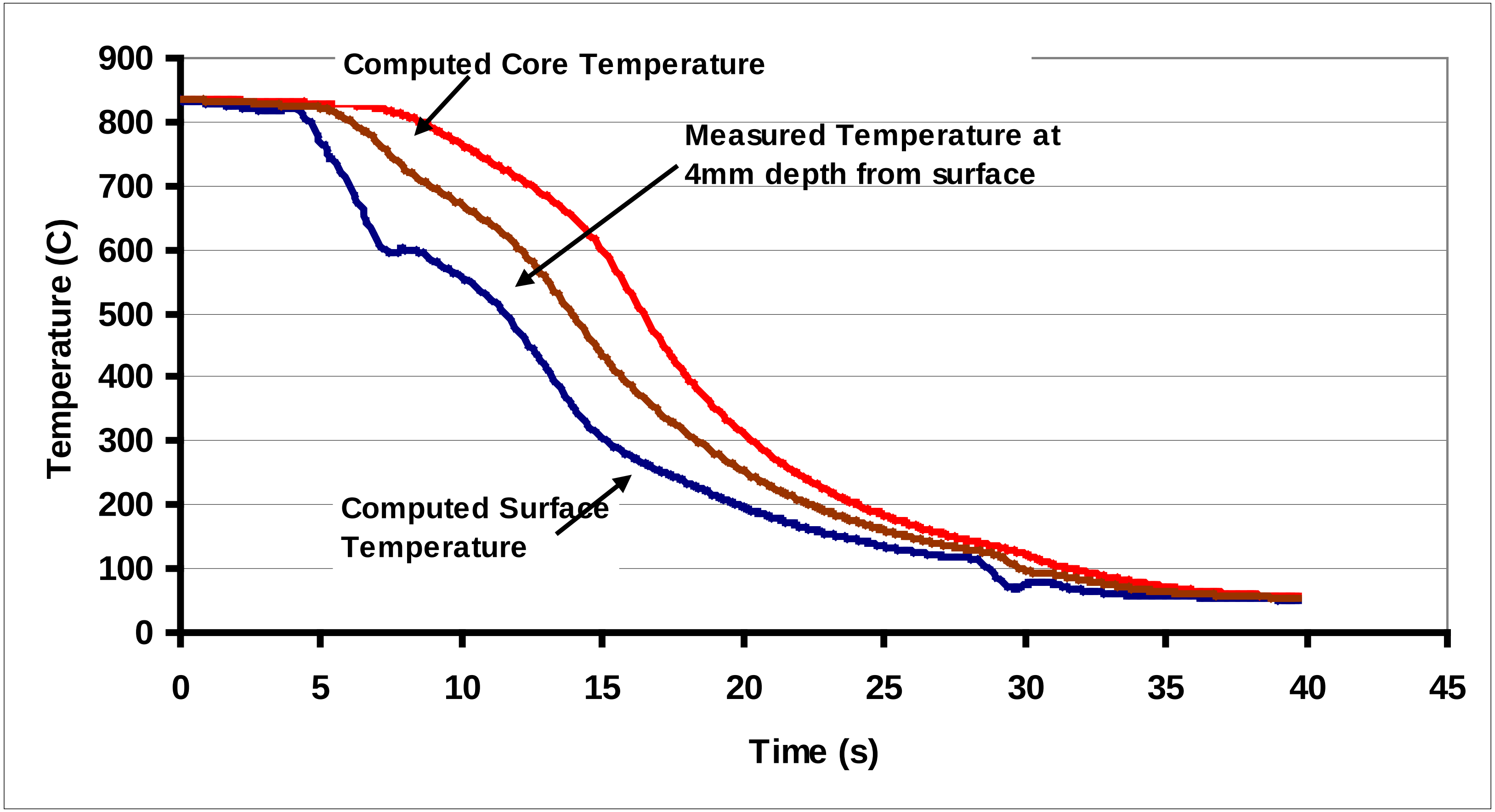



Computed surface heat flux during quenching of steel specimens of **different grades** in identical **mineral oils**

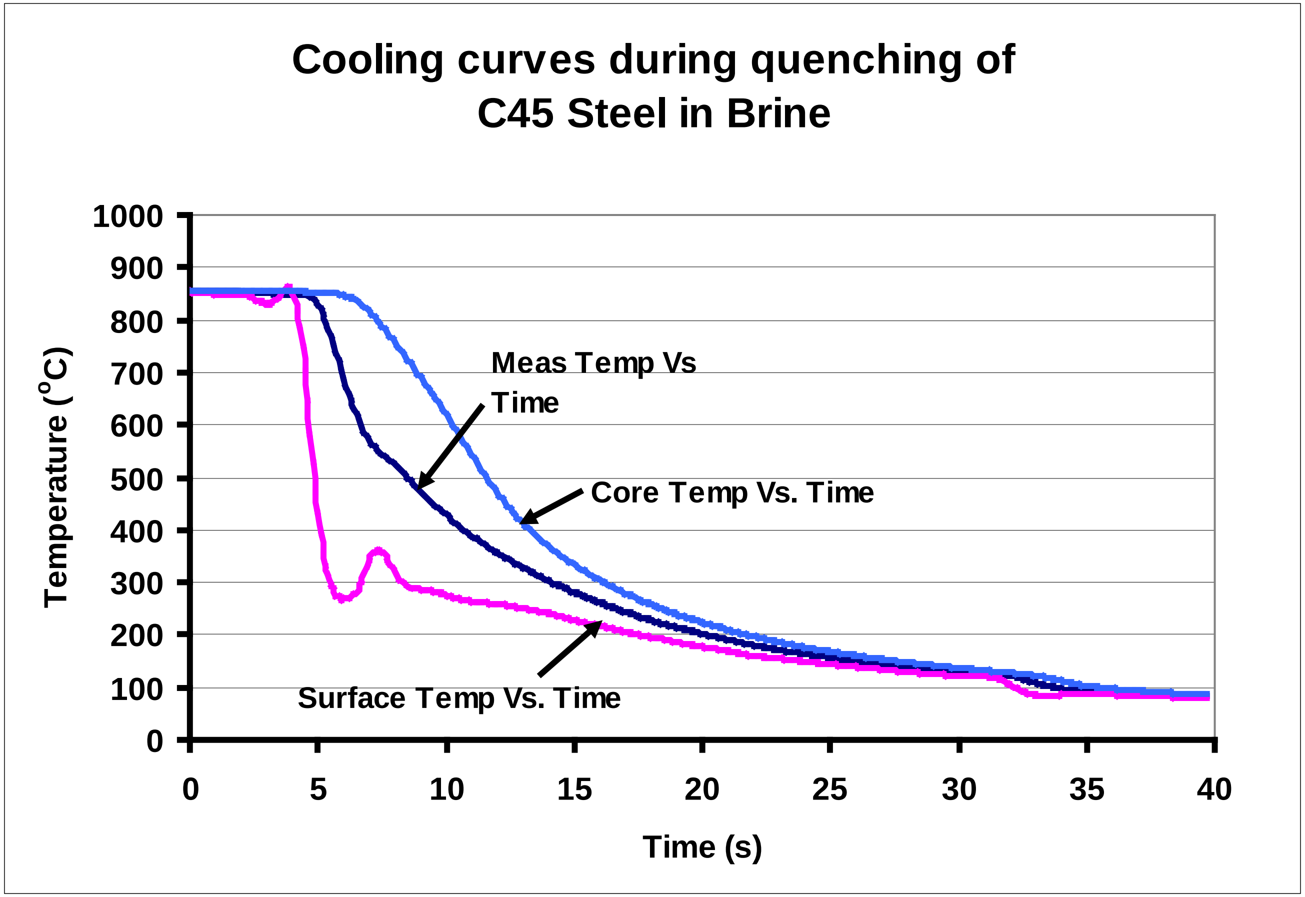



Computed surface heat flux during quenching of steel specimens of **different grades** in identical **solutions of a polymer**

# Anomalous surface heating during quenching – C45 in mineral oil



# Anomalous surface heating during quenching – C45 in brine



# “QuenchProbe” Breakthroughs

- **Test Method**:  Cooling Curve Analysis

- **Traditional Methods**: Most of the probe materials are DIFFERENT FROM THE STEEL being heat treated. The results will not be the same as when a component is quenched

- **New Method:** SAME GRADE STEEL as the component. The results are exact as when the component is quenched

# “QuenchProbe” Breakthroughs

- **Test Method**:  Hardening power of oil
- **Traditional Methods**: Indirect, statistical test. Has no direct relevance with the steel being quenched
- **New Method:** The effect of oil degradation due to any conceivable factor on heat removal is directly measured in quench tanks dynamically

# "QuenchProbe" Breakthroughs

- **Test Method**:  Effect of Viscosity, Oil contamination, oxidation, usage factor etc.

- **Traditional Methods**: Done in a lab under 'static' conditions

- **New Method:** The cooling rates are calculated for the SPECIFIC steel and quenchant combinations.

# “QuenchProbe” Breakthroughs

- **Test Method**: Data acquisition for cooling curve analysis
- **Traditional Methods**: Single thermocouple at Center of specimen, giving only ONE cooling curve for the entire sample
- **New Method:** Single thermocouple at midsection, giving multiple cooling curves at CENTER and CORE. Advanced heat transfer calculations differentiates cooling at center and surface of component.

# “QuenchProbe” Breakthroughs

- **Test Method**: Microstructure prediction
- **Traditional Methods**: Not possible
- **New Method:** Possible. Thermal analysis of the probe coupled with metallurgical transformation  for prediction of microstructure and hardness, FIRST TIME EVER.

# “QuenchProbe” Breakthroughs

- **Test Method**: Selection of quenchant for different grades of steel

- **Traditional Methods**: By trial and error based on experience

- **New Method:** Data base generated for different quenching mediums can be used of designing process sheets for new products -  selection of quenchant for optimal quality in terms of hardness distribution, stress distribution, cracking susceptibility etc.

# “QuenchProbe” Breakthroughs

- **Test Method**: Effect of agitation (circulation speed)
- **Traditional Methods**: Not possible
- **New Method:** Gives heat removal rates under plant operating conditions of agitation etc. Tests are done in-situ with a portable probe

# Applications of "QuenchProbe"

- *In-situ* measurement of quenching capacity of different quenching mediums
- Check the 'health' of quenchants with continued use
- Check the effect of agitation level/flow rate/quenching position in the tank
- Check the effect of contamination in quenchants (water in oil; polymer in oil etc.)
- Inspection of quenchants in as-received condition
- Estimate the cooling rates at the surface and core of specimen

# Applications of "QuenchProbe"

- Predict microstructure / hardness at the surface and core of alloy steel specimen for a quenchant
- Generate data for
  - mathematical modeling of heat flux for a specific plant condition
  - calculating stresses in quenched component by Finite Element Analysis
- Select the most suitable quenchant for a given component to achieve the required hardness range
- Reduce rejections and Ensure consistent quality

# Applications of “QuenchProbe”

- *In-situ* measurement of quenching capacity of different quenching mediums
- Check the ‘health’ of quenchants with continued use
- Check the effect of agitation level/flow rate/quenching position in the tank
- Check the effect of contamination in quenchants (water in oil; polymer in oil etc.)
- Inspection of quenchants in as-received condition
- Estimate the cooling rates at the surface and core of specimen
- **ALL TESTS WITH REFERENCE TO SPECIFIC GRADE OF STEEL**

# About “QuenchProbe”

- ‘QuenchProbe’ is a result of research conducted at IIT Madras in the past 12 years.

- Six research theses; four research projects (sponsored by the Indian Space and Fusion Energy Projects and others)

- Eleven International Refereed Journal papers

- Eighteen International and National Conference / Symposium / Workshop papers have been published so far, related to the technology of QuenchProbe.

# “QuenchProbe” – Industrial Tests

| Sl No | Company |
|---|---|
| 1 | Automotive Axles Ltd, Mysore, India |
| 2 | Bharath Earth Movers Ltd., KGF, India |
| 3 | Bharath Forge Ltd., Pune, India |
| 4 | HAL, Bangalore, India |
| 5 | L&T, Hazira, India |
| 6 | LVM, Bangalore, India |
| 7 | Mahindra Forge Ltd., Pune, India |
| 8 | NBC Bearings, Jaipur, India |
| 9 | SKF Bearings, Pune, India |
| 10 | SSS Springs, Siriperambudur, India |

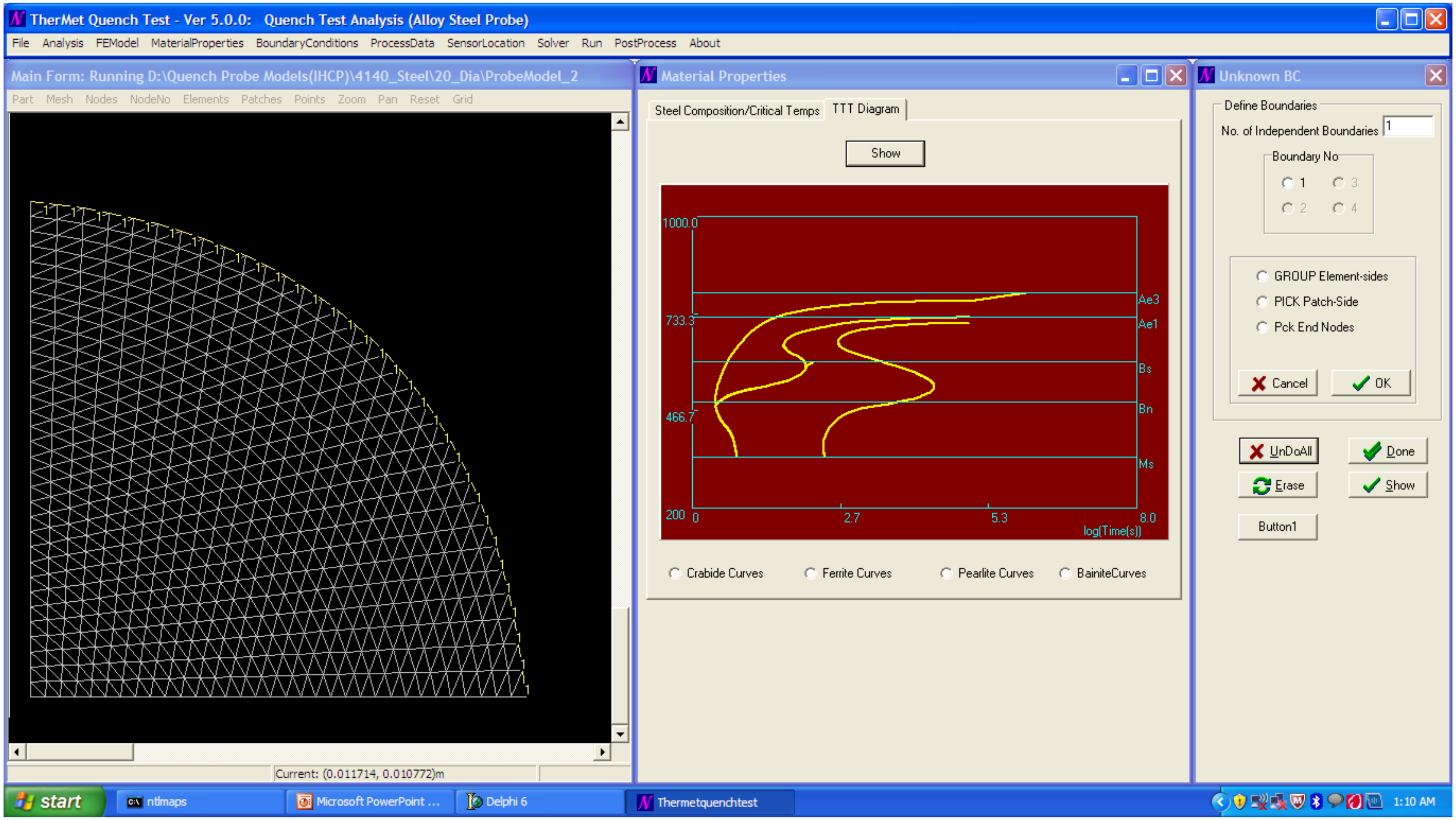
**Steels tested:**

C45, 41Cr4, 100Cr6, 8822H, SA 542, 52100, 4140, SUP 9

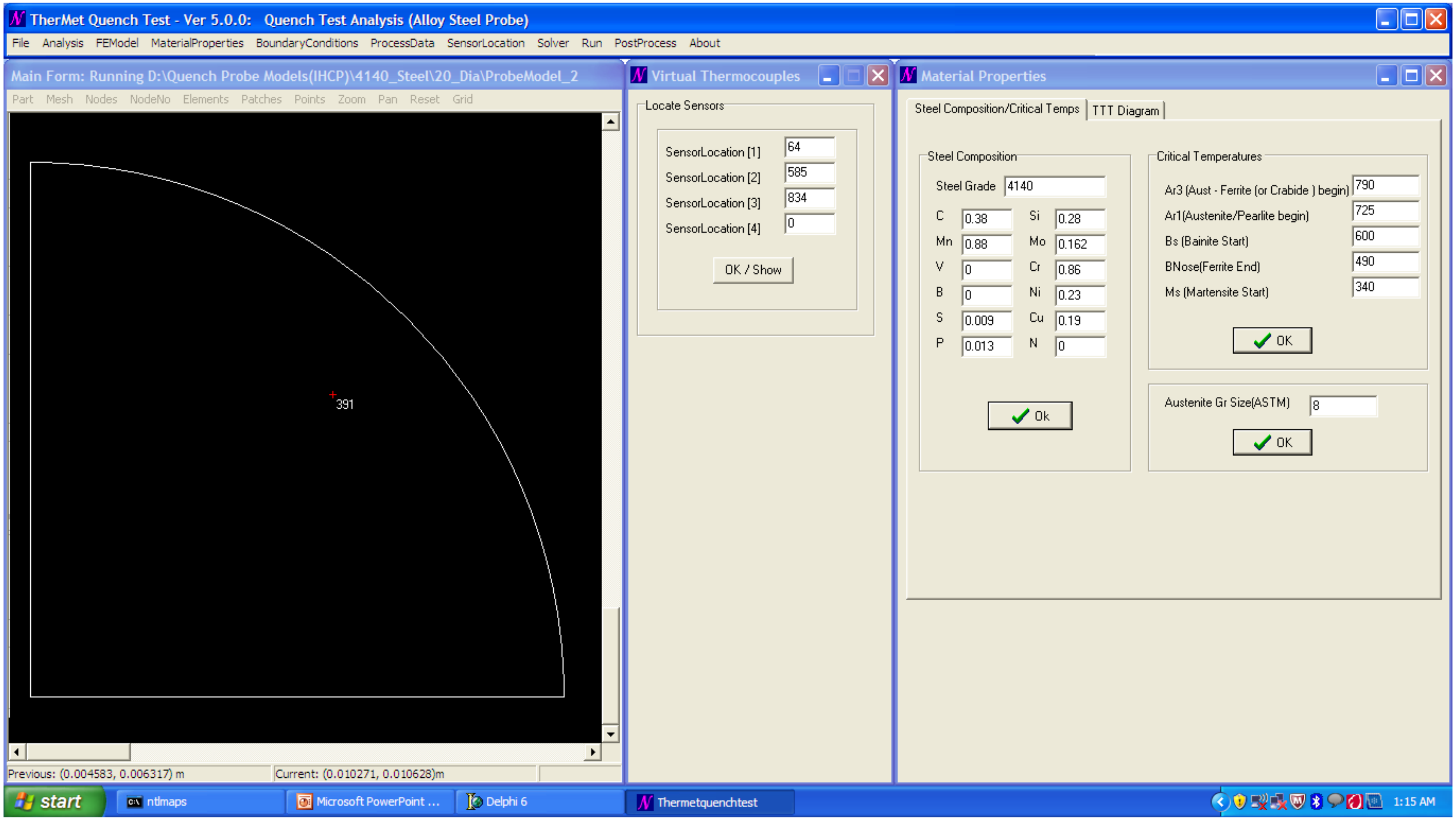
**Quenchants tested**

Servo 707, Castrol 798, Nippon 303, Hardcastle Polymer solutions (4.5%, 6.0%, 13.5%, 14.0%) **All in agitated tanks**

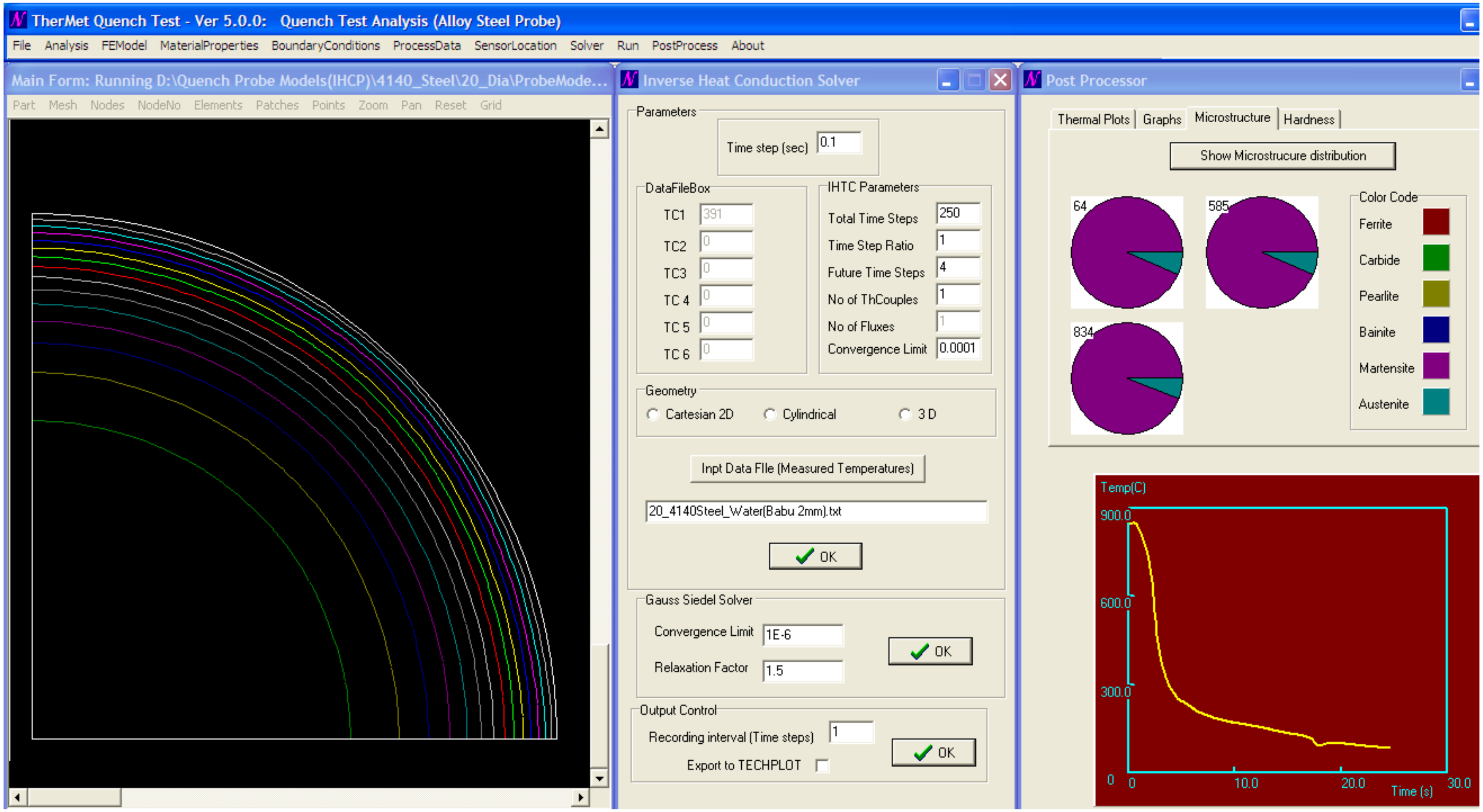
# User Interface



# User Interface



# User Interface



# To sum up…..

- Portable and handy equipment – design under patent
- Steel-Quenchant-Plant specific cooling curves
- Cooling curve obtained at 4 mm from specimen surface enables accurate heat flux estimation – the only probe of this type
- Cooling rates at the specimen surface and center can be differentiated – different diameter, user specific specimens

# To sum up…..

- Mathematical model based on experimental data base - TTT diagrams and hardness data
- Microstructure and hardness variation prediction across specimen cross section
- All software indigenously developed - ensures continuous development and tuning
- Heat flux data can be utilized for further analysis of heat treatment