Anneal Fundamentals

Improving Yield

Anneal Fundamentals: Goals of this Presentation

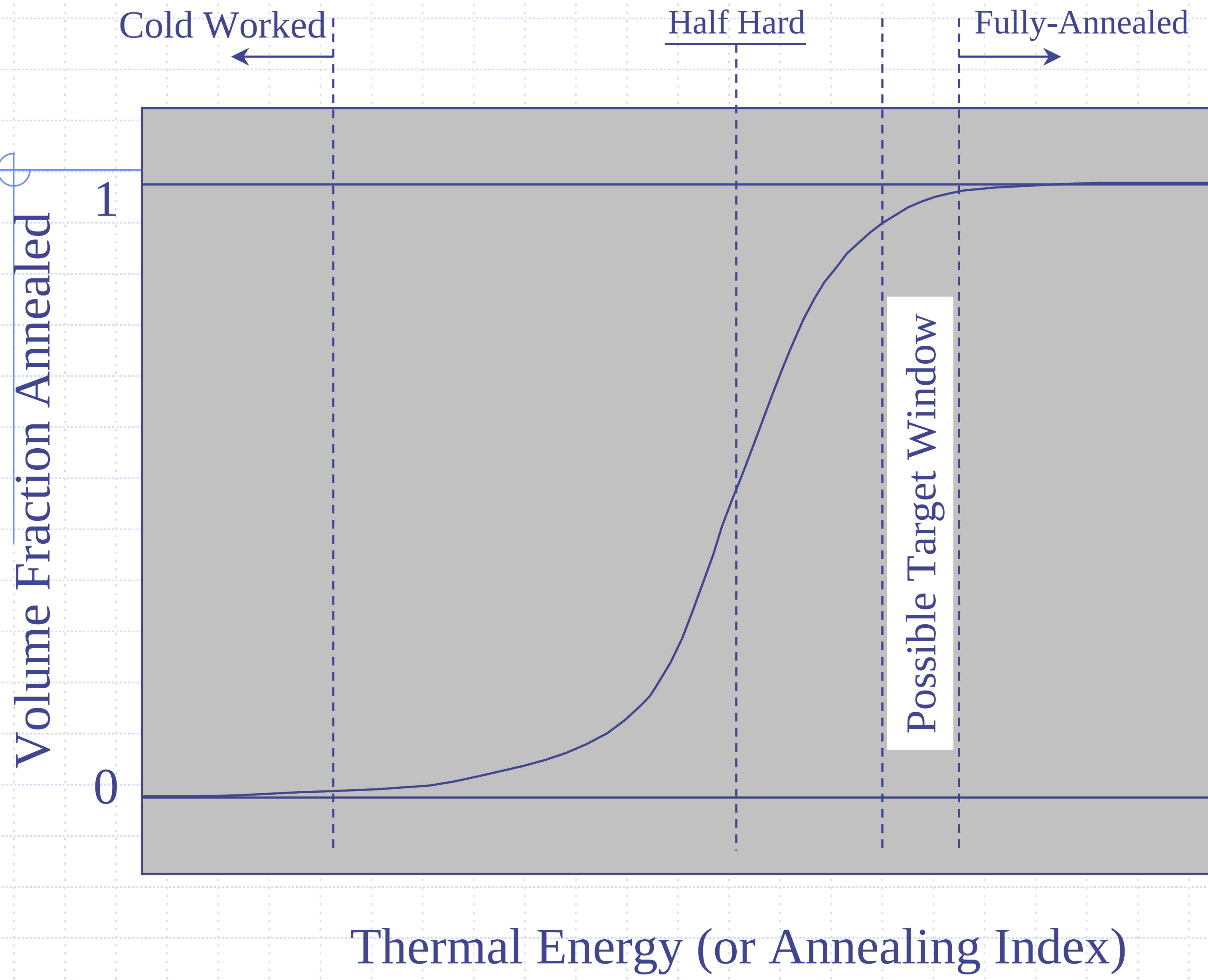
- The main goal of this presentation is to teach fundamentals of annealing that will allow for annealer control that reduces improperly annealed product and downtime.

Annealing Tutorial

- What is annealing?
- What are goals in annealing?
- What is activation energy?
- What factors influence activation energy?
- How to Set the Energy Input to An Annealer
- What is the annealing index?
- Annealability testing methods
- Annealer modeling methods
- Different Types of Annealers

What is Annealing?

- A thermal process that releases energy within metals
- It reverses the influence of cold work
- Properties follow an S-shaped (sigmoidal) curve
- Governed by the mechanisms of recovery, recrystallization & grain growth

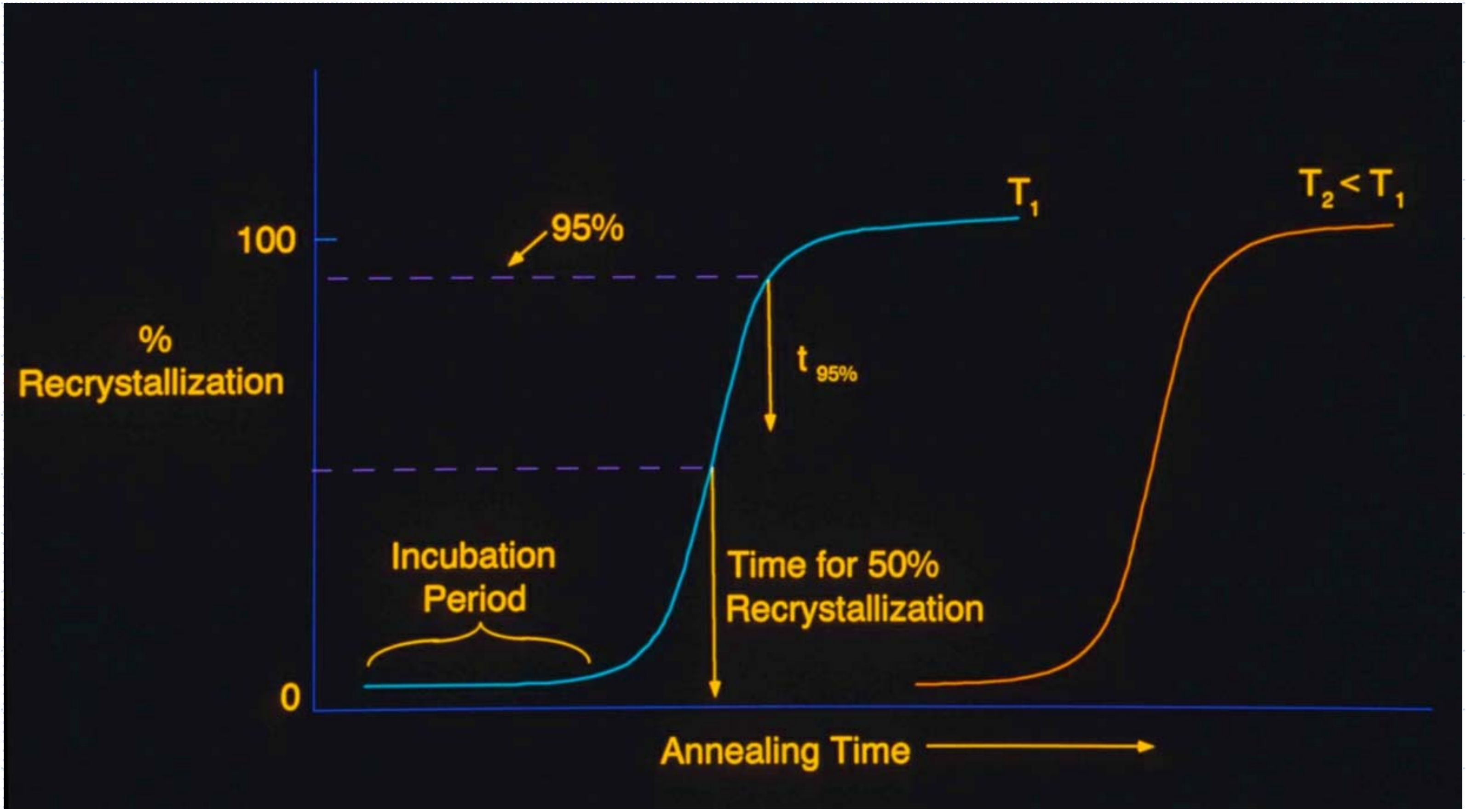


Cold Worked
Half Hard
Fully-Annealed
1
0
Volume Fraction Annealed
Possible Target Window
Thermal Energy (or Annealing Index)

What is Annealing?

- Thermal energy is added to the material until a recovery phase begins, which is followed by recrystallization of the grains.
- The material properties only start to make permanent change when recrystallization starts.
- Before recovery is an incubation phase where no significant property change occurs.
- Different materials begin recovery at different energy levels.

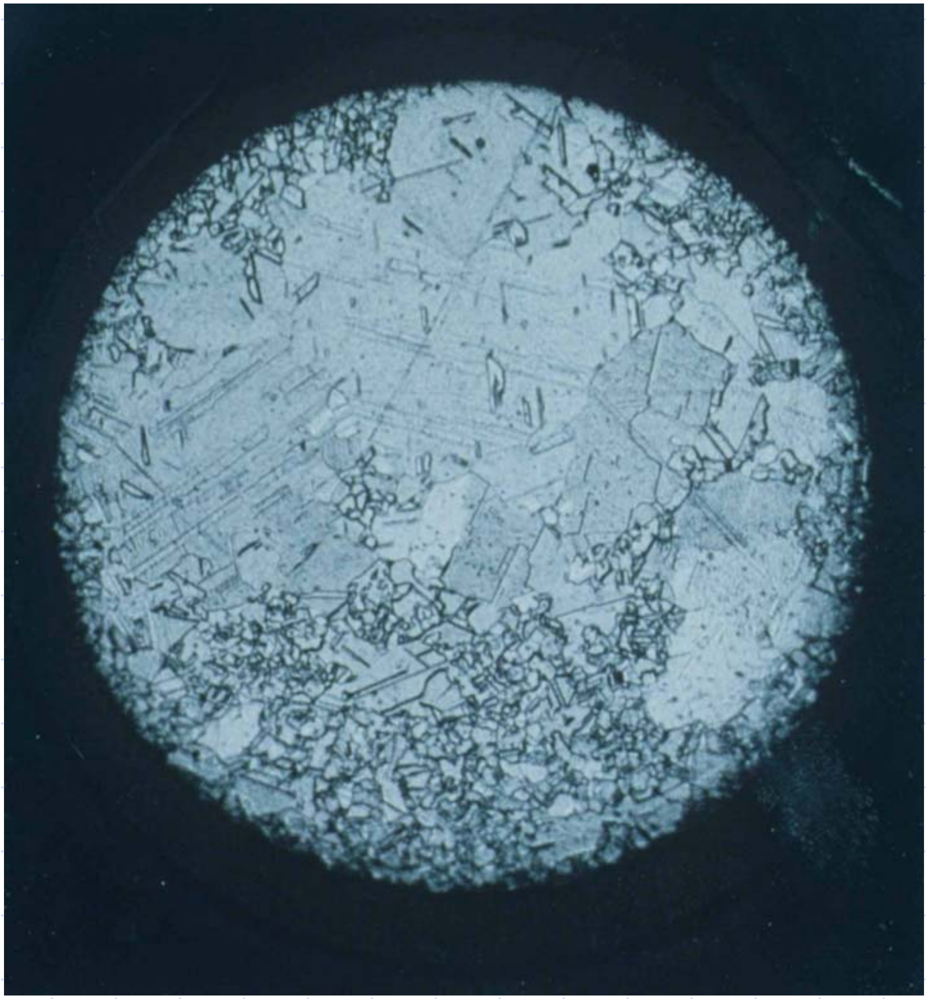
Recovery & Recrystallation at Different Energy Levels



Goals

- Some annealing processes must aim for a target window along the S-shaped annealing curve. Knowledge of the material and of the annealer is crucial to ensure product witnin this target window every time.
- Other annealing processes are less sensitive and may just require a “fully annealed” condition to the far right of the S-shaped annealing curve. However, in some cases, there is still a danger of over-annealing, which can cause abnormal grain grow leading to lumpy product or stretching in post processing.

Abnormal Grain Growth



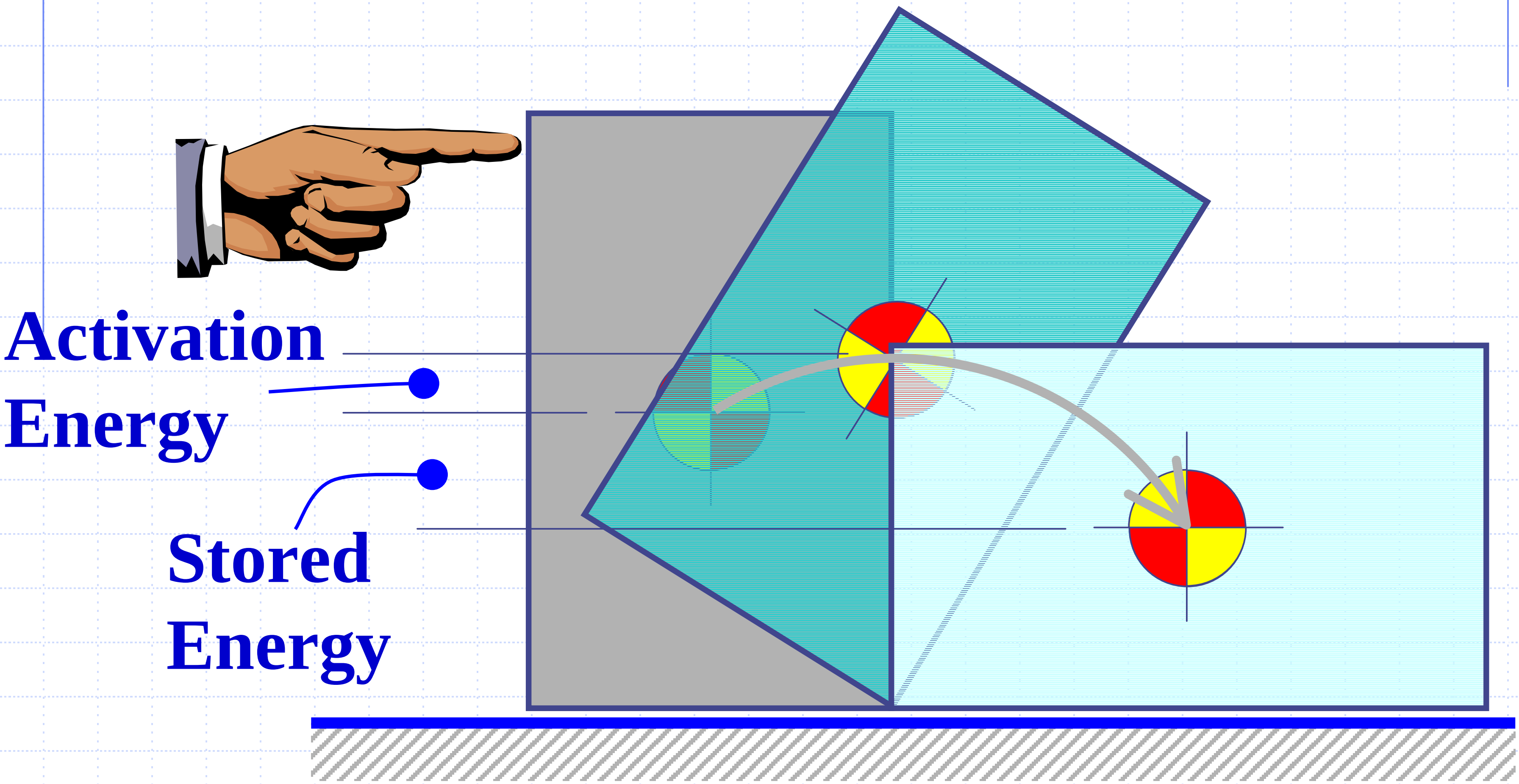
Activation Energy, Q

- As the Activation Energy, Q, increases more energy is required to anneal the metal and it is said to be more "sluggish" in annealing
- Softening Rate = 1/t = A $e^{-Q/RT}$
- To increase energy, either the the processing time and/or the processing temperature(s) must increase.
- Q is a function of the quantity and state of impurities and the amount of cold work
- For ETP Cu. Q = 35 kcal/mol – 2.5×10^{-3} O_2

Activation Energy, Q

- To cause the start of recovery and recrystallization the thermal energy must overcome the activation energy of the material to release the store energy in the material.
- The stored energy had been added to the material during deformation.
- Activation energy decreases as prior cold work increases.

Mechanical Analogy of the Activation Process



What Factors Influence Activation Energy, Q?

- Quantity of Impurities
- State of Impurities
- Rod Casting and Hot Working Parameters
- Redundant Work
- Texture
- Drawing Temperature
- Handling
- Wire Size

State of Impurities

- Impurities can be dissolved or undissolved in the solid (parent matrix)
- Dissolved impurities tend to raise both the recrystallization temperature and the activation energy when they are making the metal more sluggish.
- Undissolved impurities have a negligible influence, because they take the form of metal oxides or intermetallic compounds

State of Impurities

- Impurities have an extremely large influence in commercially pure metals.
- Scavenging elements can be added to metals to draw impurities out of solution. For examples:
  - Oxygen in ETP copper
  - Boron in EC aluminum
- The percentage of impurities forming particles is a function of the thermo-mechanical processing history

Rod Casting and Hot Working Parameters

- Faster casting and hot working (i.e. hot rolling) produces more sluggish annealing rod and drawn wire.

Redundant Work

- Redundant work increases with increasing die angle and decreasing % reduction/pass
- It also increases with asymmetric drawing
- Redundant work add more strain energy to the metal, but this added energy has a negligible influence on annealing response
- However, redundant work also alters the texture of the wire causing significant changes to annealing

Texture

- The wire texture formed during drawing is influenced by
  - Percent Reduction
  - Redundant Work
  - Drawing Temperature
- Certain wire textures anneal faster than others

Other Factors Influencing Q

- • Drawing Temperature: Higher temperatures can reduce the amount of strain energy and therefore retard subsequent annealing.
- • Handling: Excessive strain in bending can work-harden soft annealed wires, but decrease the yield strength of hard drawn wires.
- • Wire Size: As wire size increases more energy must be provided to reach required temperatures due to increased mass and often reduced efficiency in absorbing energy.
- Strain-Rate: Appears to have little influence on annealing response.

What is the Recrystallization Temperature?

- For an equivalent time period, different materials start recrystallizing at different temperatures, called the Recrystallization Temperature.
- In high-speed annealers, the recrystallization temperature is only useful as a comparison of materials.

Approximate Recrystallization Temperatures of Various Materials

Material (1 hour)	**R.Temp,ºF**	**RT / Melt Point**
Aluminum (99.999%)	180	.38
Aluminum (99.0%)	550	.60
Copper (99.999%)	250	.29
Copper(O_2 Free)	400	.35
Iron	850	.39
Lead	50	.43
Nickel	1100	.34
Silver	400	.38
Tin	50	.50

How to Set the Energy Input to An Annealer

- Determine the acceptable range of mechanical or electrical properties required
- This range will correspond to a section of the S-Shaped Curve of Slide #3
- Many use trial and error to set adjust the annealer settings until the desired properties are observed.
- However, trial and error is costly (scrap) and it does not develop insights into the process

How to Set the Energy Input to An Annealer

- A more disciplined approach is to determine the annealing index of the typical process material
- Then set up the annealer to produce that annealing index in the process

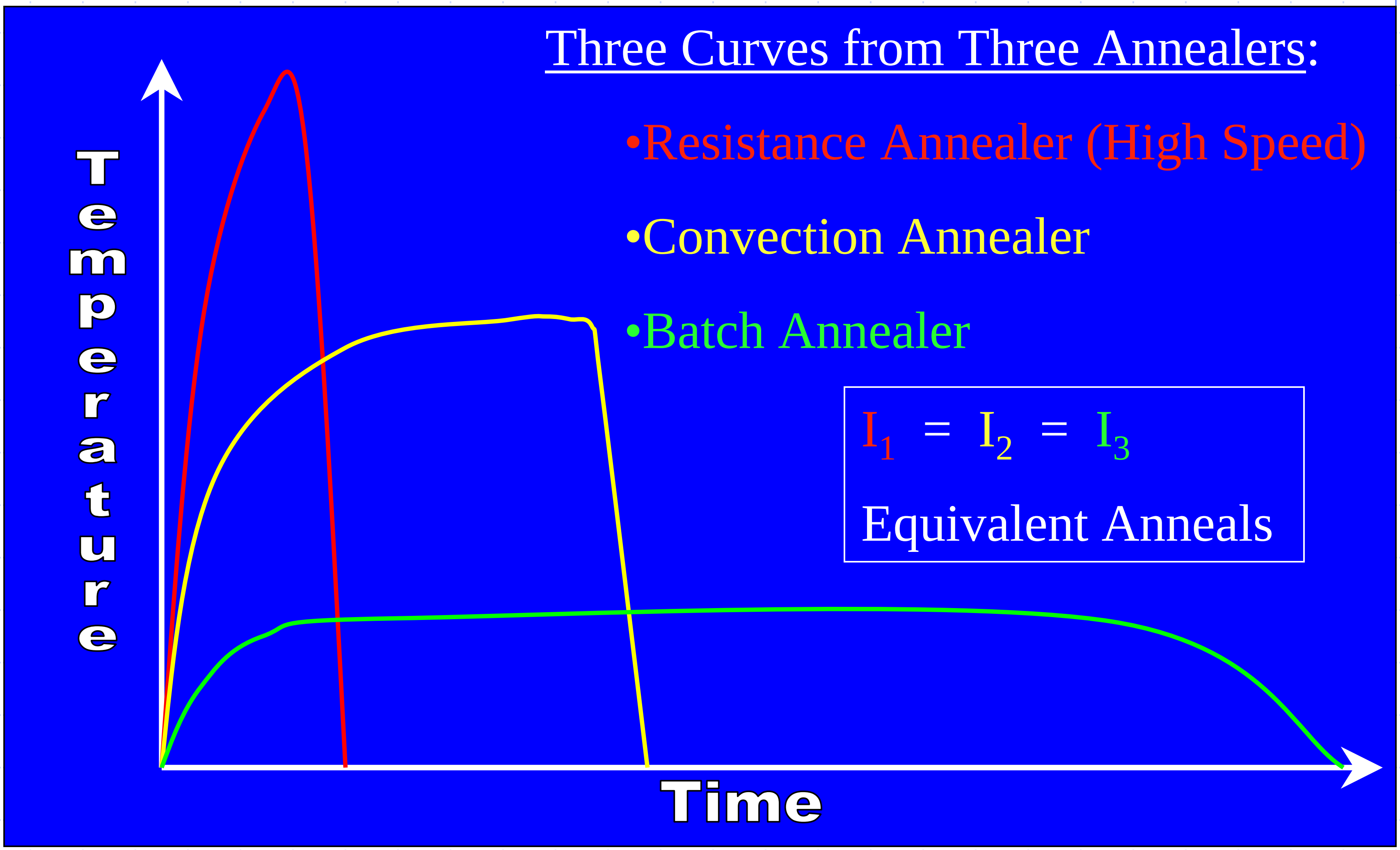
Annealing Index, I

- The energy input into metal to overcome the activation energy, Q, has two dimensions: Temperature and Time
- The annealing index, I, equates equivalent annealing processes performed at differing temperatures and times
- Time may be dictated by outside processes
- The index can be calculated by the integral below or by a summation for computer analysis

$$I = \frac{1}{2.303} \ln \int_0^{t_f} e^{-Q/RT(t)}\, dt$$

From Reference #1:
Kraft, Wright & Jensen

Annealing Index, I, Equates Processing from Various Annealers



Annealability Testing: How to determine the Annealing Index of a Metal

- It is important to determine the proper annealing index (I) for each material. To do so the following information is needed:
  - The desired annealed property range
  - The activation energy (Q) of material
- To determine Q an annealability test must be performed.

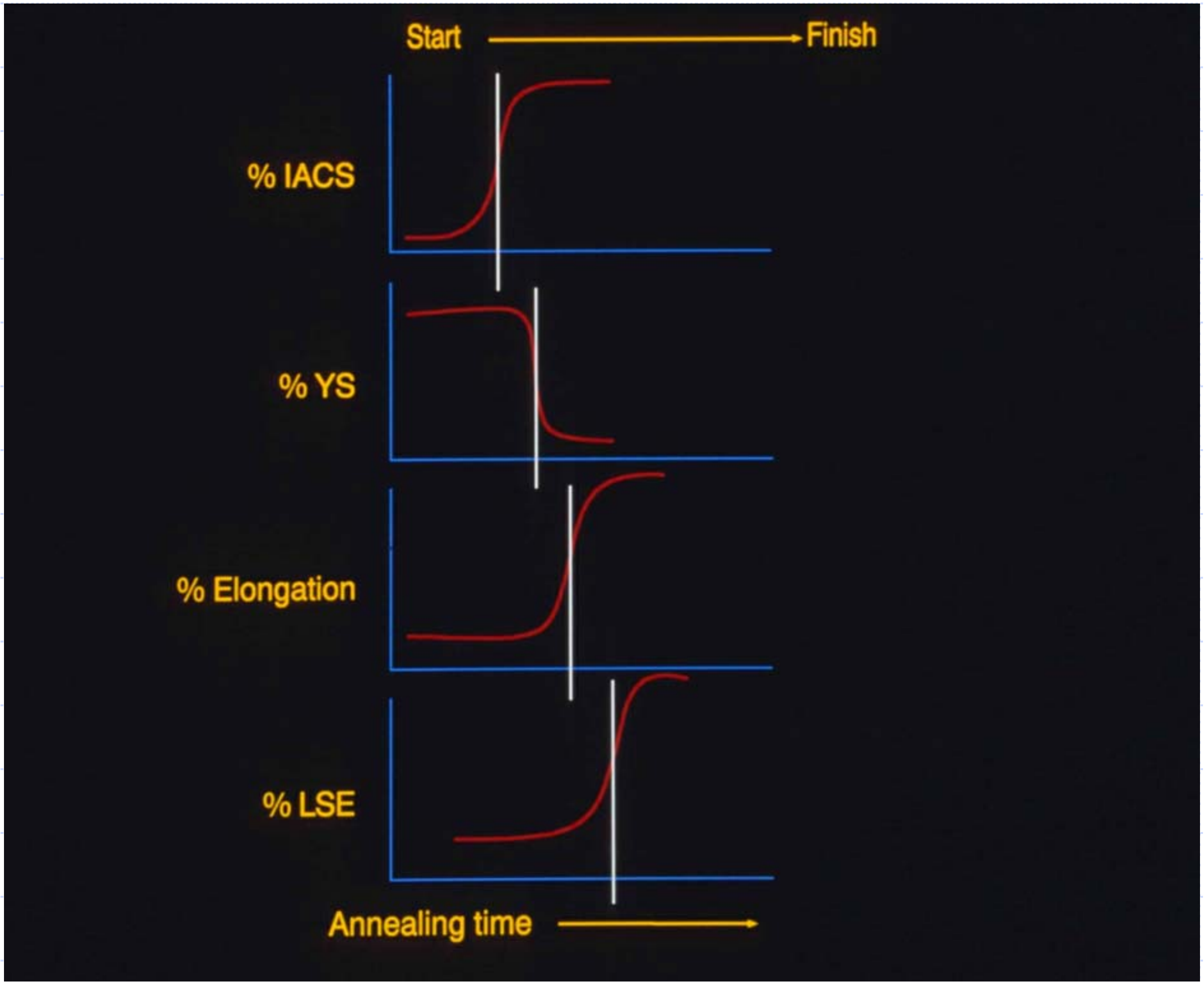
Annealability Testing:

- Annealability tests can be broken up into three categories:
  - Traditional: Exhaustive but slow tests
  - Fast but limited tests
  - A combination of fast and exhaustive tests
- The main problem with all the <u>fast</u> tests is they do not <u>directly</u> measure the properties that are desired in the annealed product.

Sequence of Property Change During Annealing

- During recrystallization multiple material properties change, but not simultaneously.
  - Ultimate tensile strength (UTS) starts and completes its transition first,
  - then metallurgical grain structure,
  - then yield strength,
  - next percent elongation by tensile test
  - and last of the five: low stress elongation (LSE).
- Yield strength shows the greatest percentage change and is considered the most sensitive.
- since LSE responds last, it should be used as a measure for ensuring full anneals.

Sequence of Property Change



Exhaustive Annealability Tests

- These tests involve heating samples in constant temperature ovens or salt bath for varying lengths of time
- The first goal is to determine the time range at which recrystallization takes place (the S-Shaped Curve). Thus, first time periods are widely spaced
- Subsequently, multiple samples are processed at multiple points along the S-Shaped Curve
- After enough samples are processed to define the curve, these samples are tested for the critical property (i.e. yield strength, LSE, Spiral Elongation, etc.)
- These tests are typically performed in a laboratory

Exhaustive Annealability Tests

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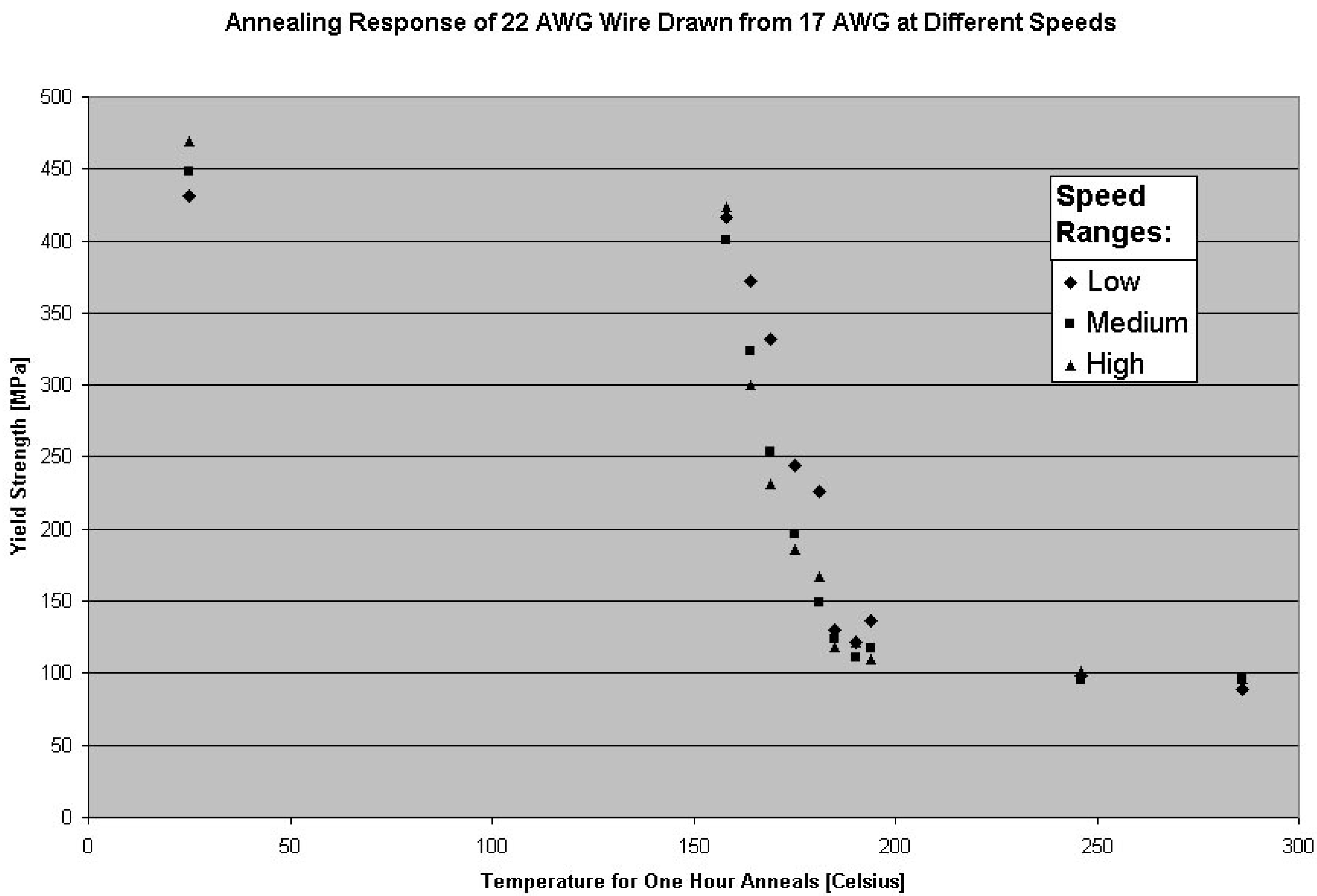
Example of Exhaustive Annealing Test Data:



Fast but Limited Tests

- While the Slow Annealability testing usually requires 4 to 8 hours of work and dozens of samples, there are tests that can provide results within 5 to 30 minutes and require only one sample
- Two fast annealability tests are:
  - Calorimetry
  - A test developed by RT Design

Calorimetry

- A calorimeter has a nearly adiabatic chamber into which the test sample is placed. Heat is added to this chamber at a constant rate while the chamber temperature is monitored very accurately. The result is a plot of time (or total heat input) versus temperature that allows one to determine at what temperatures material changes take place. This method is most commonly used to determine phase changes of any materials and it is more often used for diagnosing non-metals.
- Calorimeters are expensive laboratory instruments
- Relatively small sample sizes are used and thus the test is , thus cutting the sample must be done in such a way that the cutting method does not introduce deformation that would influence the test

Calorimetry

- Advantages:
  - Calorimetry is very fast and automatic.
- Disadvantages:
  - Calorimeters are expensive laboratory instruments that are often not designed to operate on the plant floor.
  - Relatively small sample sizes are used and thus the test can be influenced by localized abnormalities.
  - Sample preparation must be performed meticulously. Thus cutting the sample must be done in such a way that the cutting method does not introduce deformation that would influence the test. Also no significant contamination should be introduced.

RT Design Annealability Tester

- A fast annealability tester has been developed that works under different principles than calorimetry and has several advantages over calorimetry:
  - It is much less expensive than the cheapest calorimeter, but has similar or better accuracy (depending on the comparison calorimeter)
  - Longer samples can be tested and sample cutting methods do not influence the test
  - It is faster than the typical calorimetry tests and thus better simulates fast production annealers
  - This test can be performed on the plant floor
  - This test can be performed on standing wire without cutting out the a sample. The length tested will be annealed, but for many processes this is acceptable
  - It is designed specifically for rod and wire, thus test set up is more convenient and results output are in the form of time, temperature, annealing index and power input
  - It is also designed to aid in the modeling of annealers (subsequent slides)

A Combination of Fast and Exhaustive Annealability Tests

- Using both fast and slow tests the benefits of both can be achieved
- First a single unknown sample is analyzed by a fast test to determine the range of Annealing Indices where the recrystallization takes place
- With this information, the second phase of the slow test method is used to clearly define the desired properties along the S-Shaped Curve

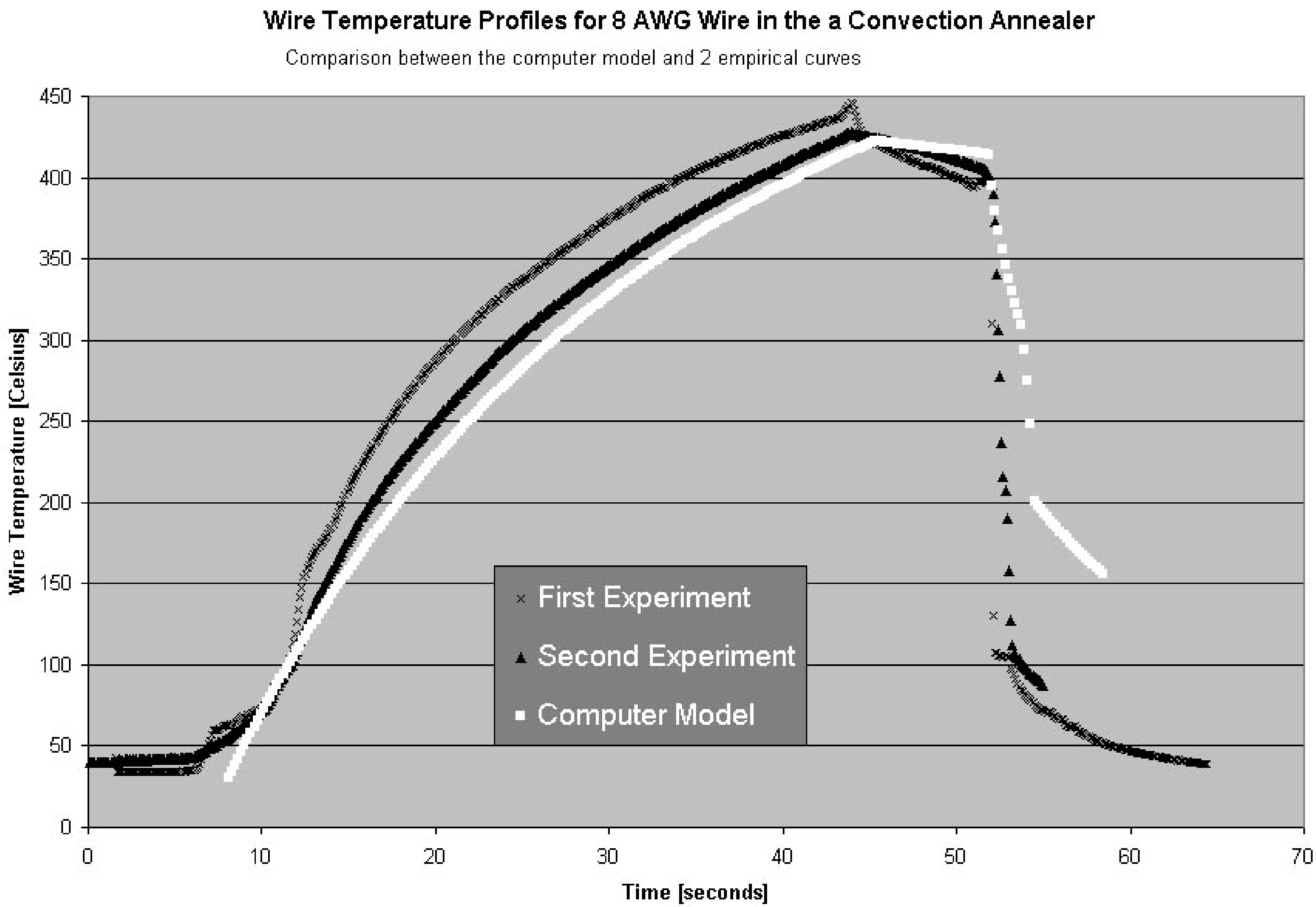
Setting an Annealer to the Desired Annealing Index, I

- Annealers are controlled by adjusting the energy input and in few instances there is minimal thermal feedback of the actual work piece
- Thus to set an annealer to an annealing index the following must be performed:
  - A theoretical model of the annealer must be created
  - An empirical model of the annealer must be measured
- These analyses are not difficult and only have to be performed once

Theoretically Modeling the Annealer

- The annealer manufacturer may have an existing theoretical model and may be willing to provide it to the customer
- RT Design can create a theoretical model, including a user-friendly interface to facilitate plant engineer's ability to run numerous "What if ... ?" scenarios
- Mathematically the finite difference and lumped capacitance method can typically be used for wire modeling
- The model must be verified by empirical data

Empirically Verifying a Theoretical Model



Empirically Modeling the Annealer

- Temperature Profile Recording using Embedded Thermocouples
- Temperature Monitoring:
  - Monitoring of Line Locations
    - Non-contact monitors
    - Temperature Sensing Sheaves
  - Monitoring of Maximum Product Temperature
    - Temperature Sensitive Paint

Different Types of Annealers

- Resistance
- Induction
- Convection
- Radiation
- Batch
- Plasma

References

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