

# **POSITRON ANNIHILATION SPECTROSCOPY IN MATERAILS STRUCTURE STUDIES**

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# OUTLINE

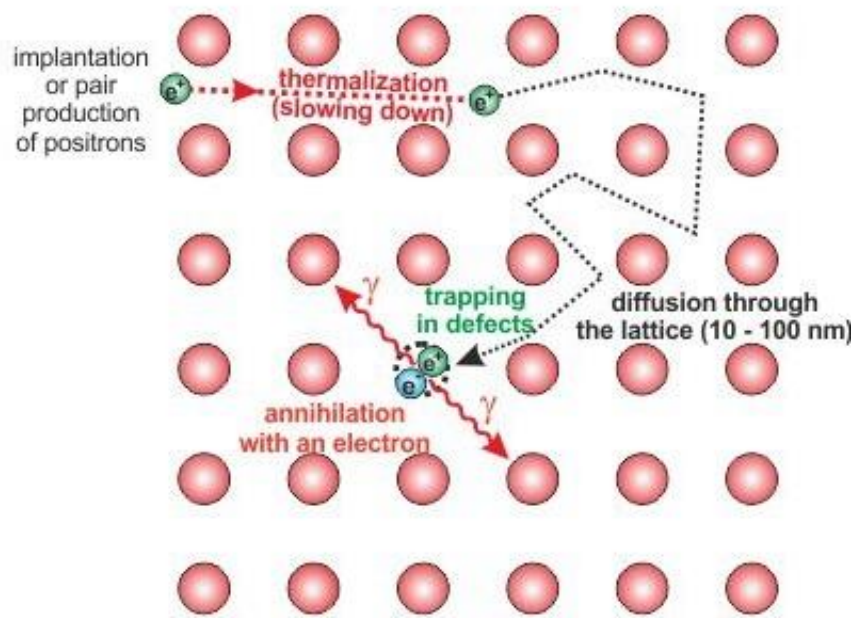
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# AIM

- ❖ To study the Recovery and Recrystallization process in pure Titanium.

# INTRODUCTION

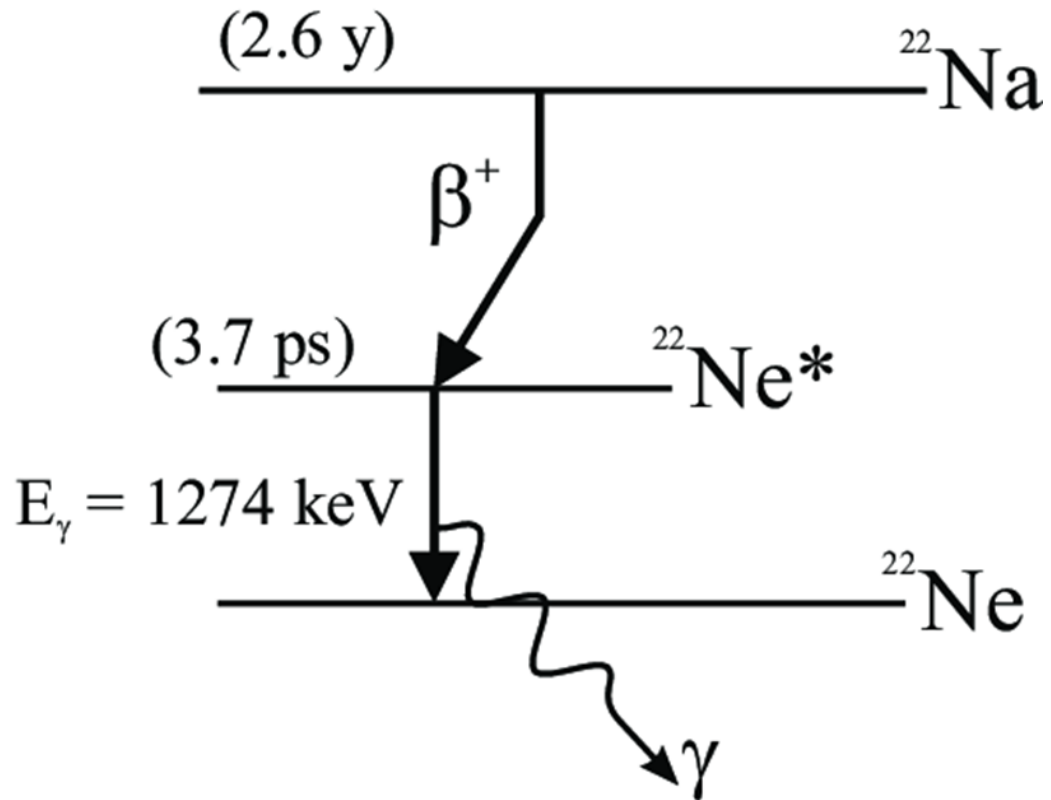
## METHODS OF POSITRON ANNIHILATION SPECTROSCOPY



- **Positron Lifetime Spectroscopy** -measures the elapsed time between the implantation of the positron into the material and the emission of annihilation radiation.

**Figure 1: Methods Positron annihilation Spectroscopy**

# Na-22 DECAY SCHEME

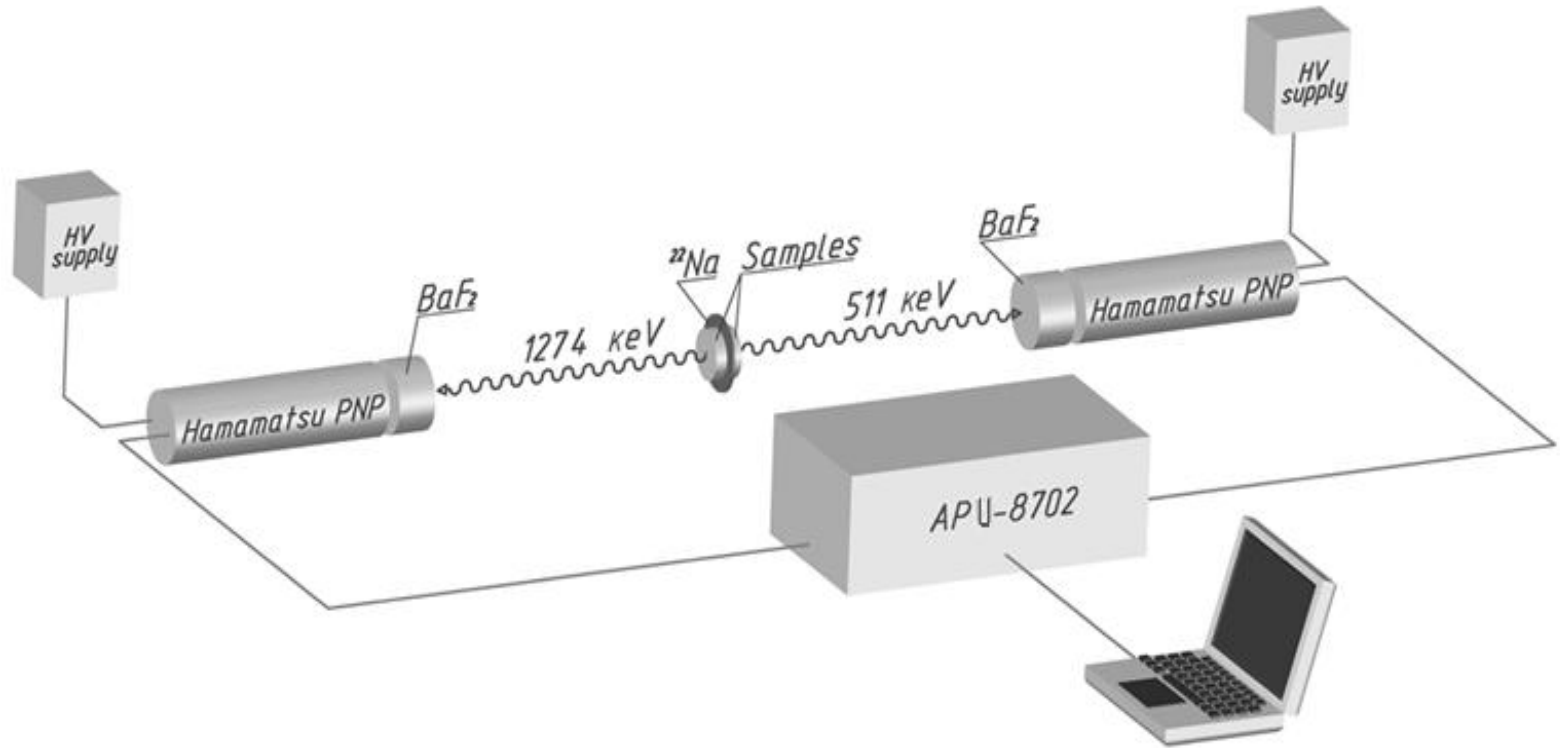


- Is emitted in the  $\beta^+$  decay of  $^{22}\text{Na}$
- $\beta^+$  decay  $\rightarrow$   $^{22}\text{Na} + \beta^+ + \nu_e + \gamma$  ( $1.27 \text{ MeV}$ )

**Figure 2: Decay scheme of the radioactive isotope Na-22.**

# POSITRON LIFETIME TECHNIQUE

- **Positron annihilation lifetime spectroscopy** is a non-destructive spectroscopy technique to study voids and defects in solids.
- - Positron lifetime is measured as time difference between 1.27 MeV quantum ( $\beta^+$  decay) and 0.511 MeV quanta.



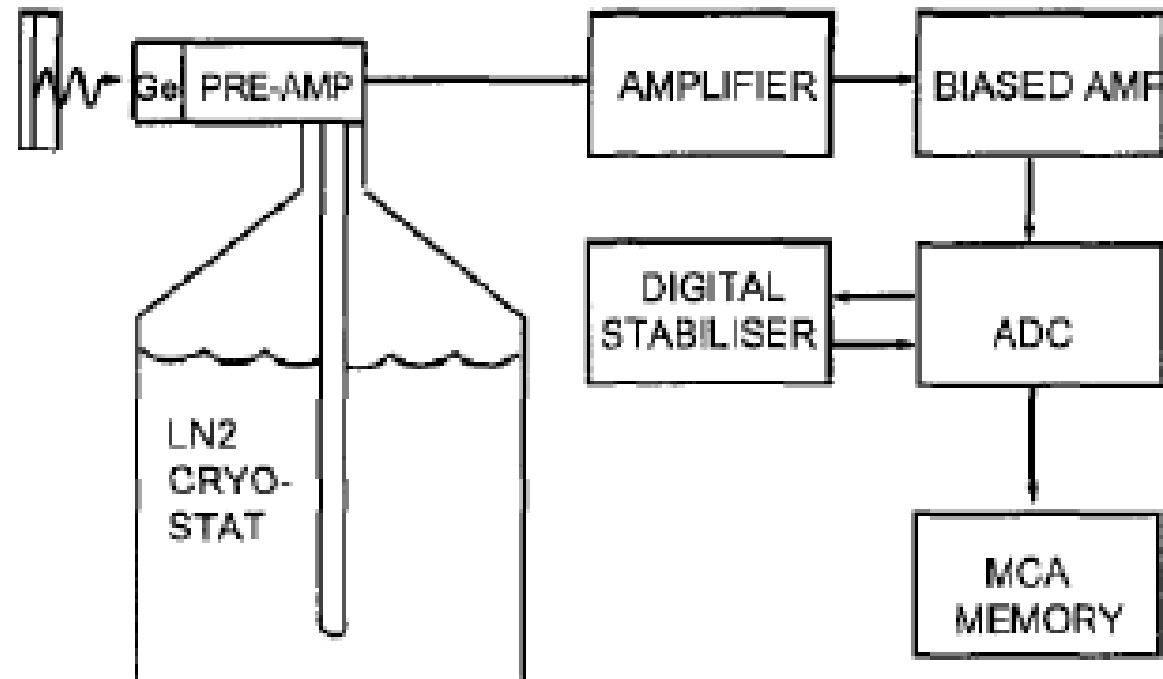
**Figure 3: Positron annihilation lifetime Spectroscopy.**

# DOPPLER BROADENING SPECTROSCOPY

- The electron momentum in propagation direction of 511 KeV  $\gamma$ -rays leads to doppler broadening of annihilation line.
- This can be detected by Ge detector and standard electronics.

$$E_g = mc^2 + E_B \pm \frac{P_L C}{2}$$

$E_B$ - binding energy of electron-positron pair



**Figure 4: Doppler broadening Spectroscopy Setup.**

# METHODOLOGY

## SAMPLES PREPARATION

- Titanium samples were polished with waterproof abrasive paper.
- The samples were cleaned with pure Acetone to remove dust and any surface contamination.
- All samples were annealed 850 °C to remove defects.



# PRESSING PROCEDURE

- The samples were pressed under a static pressure from 4.5 to 11.5 MPa for 1 minute at room temperature.
- The thickness of the samples were measured using Micrometer screw.



**Figure 6: Micrometer Screw**



**Figure 5: Hydraulic press**

# ANNEALING

- The annealing treatments were carried out in a vacuum  $10^{-4}$  Pa with temperature being controlled.
- The series of samples were heated at the temperature range from 100-850°C for 1 hour.



Molecular pump

Figure 7: Vacuum system.

Furnace

# POSITRON LIFETIME SPECTROSCOPY MEASUREMENTS

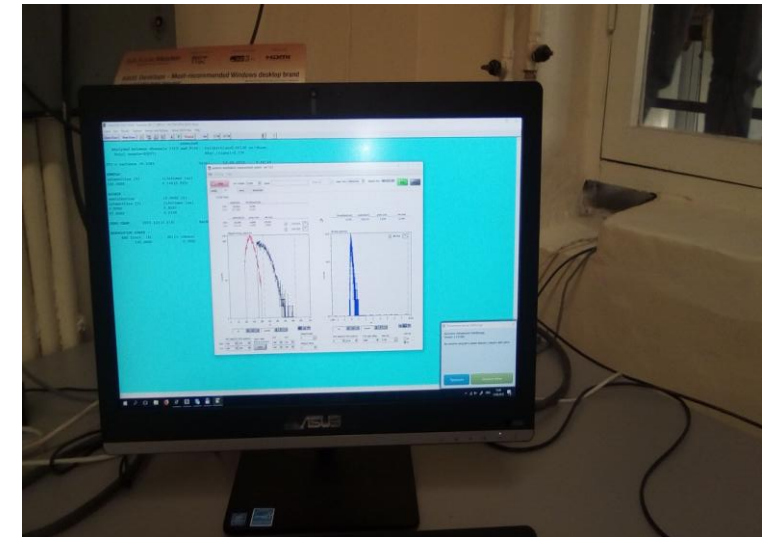
- The  $^{22}\text{Na}$  were placed between the identical Titanium samples and attached to two photomultiplier and computer display.
- The samples were analysed for 3 hours.



ADC



Titanium samples attached to two photomultipliers.



Computer display

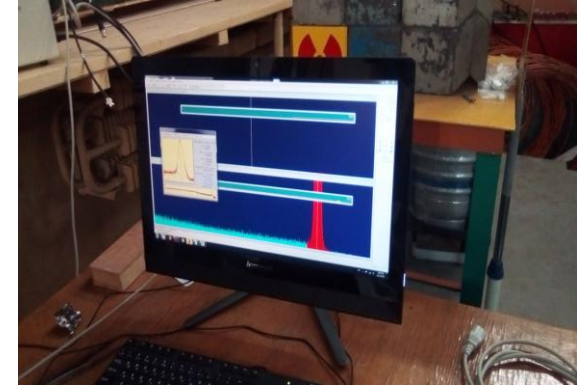
Figure 8: Positron Lifetime Spectroscopy setup.

# DOPPLER BROADENING SPECTROSCOPY MEASUREMENTS

- The samples were placed near the high-purity(HP)-Ge detector and standard electronics with energy resolution of about 1.2 KeV at 511 KeV.
- The samples were analysed for 2 hours in the detector system.



**Liquid Nitrogen**



**Computer display**



**Power system**



**Titanium Sample( DBS-Stage)**

**Figure 9: Positron Doppler Broadening Spectroscopy Equipment.**

# S- AND W- PARAMETERS CALCULATIONS

$$E_g = mc^2 + E_B \pm \frac{P_L C}{2}$$

- $E_B$ - binding energy of electron-positron pair

## S-Parameter

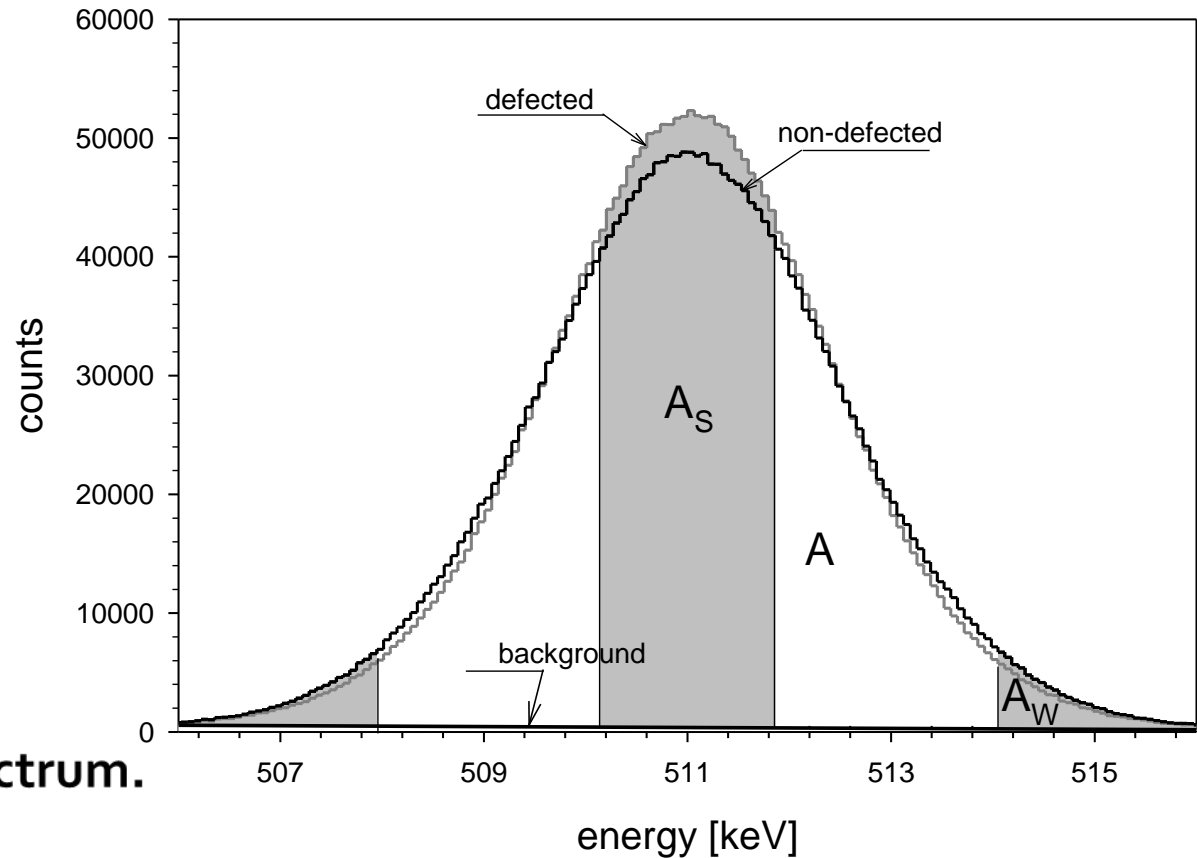
- Relation of area under central part of annihilation line  $A_S$  to total area below this line  $A$ .

$$S = \frac{A_S}{A_0}$$

## W-Parameter

- Sum of the area under wings of the spectrum.

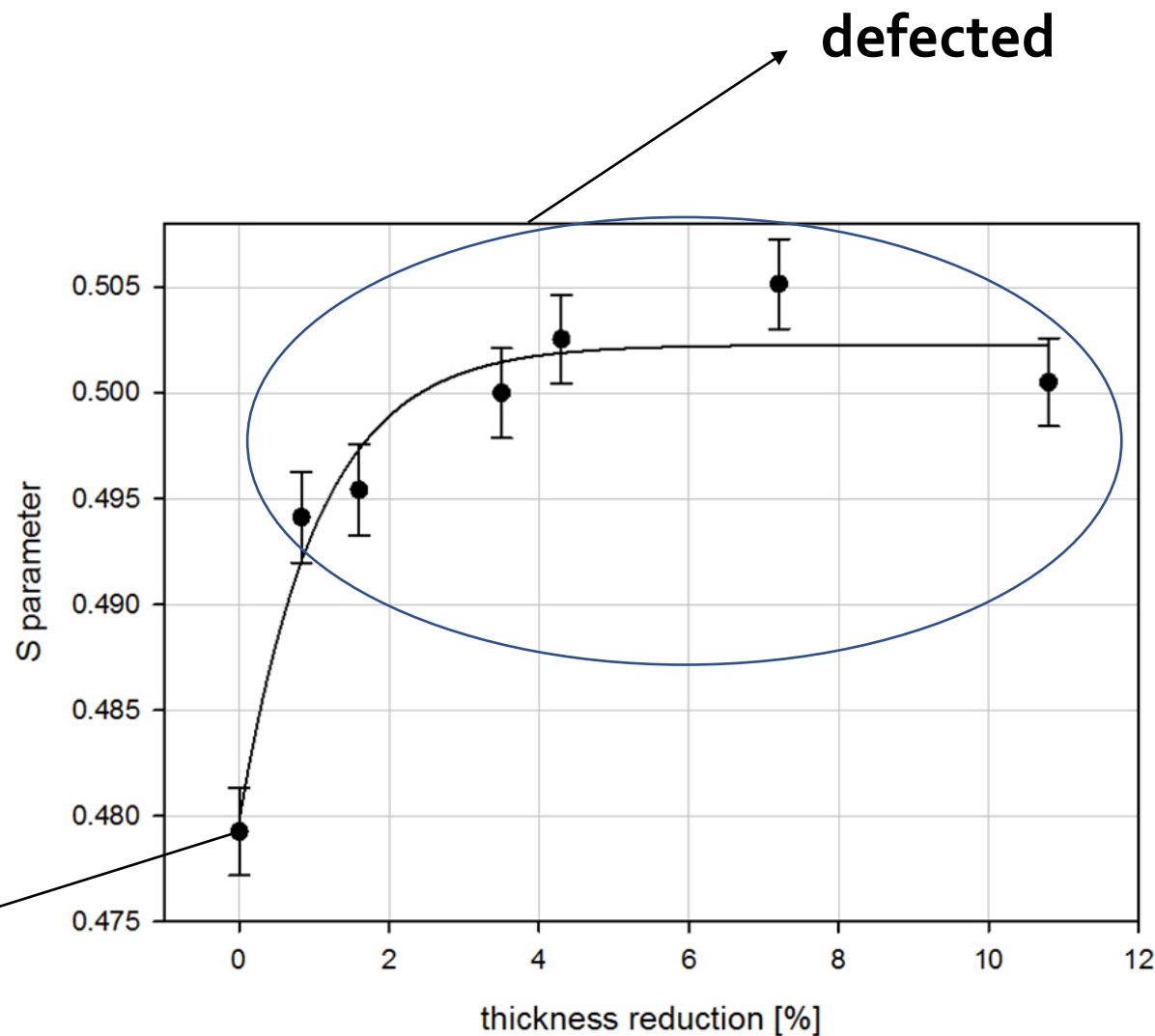
$$W = \frac{A_W}{A_0}$$



**Figure 10: Comparison of annihilation for defected and non-defected samples. The rule of calculation of S-and W-parameters**

# THICKNESS REDUCTION

- The pressing procedure increases number defects on the sample.
- The S-parameter increases with the thickness reduction of the sample.



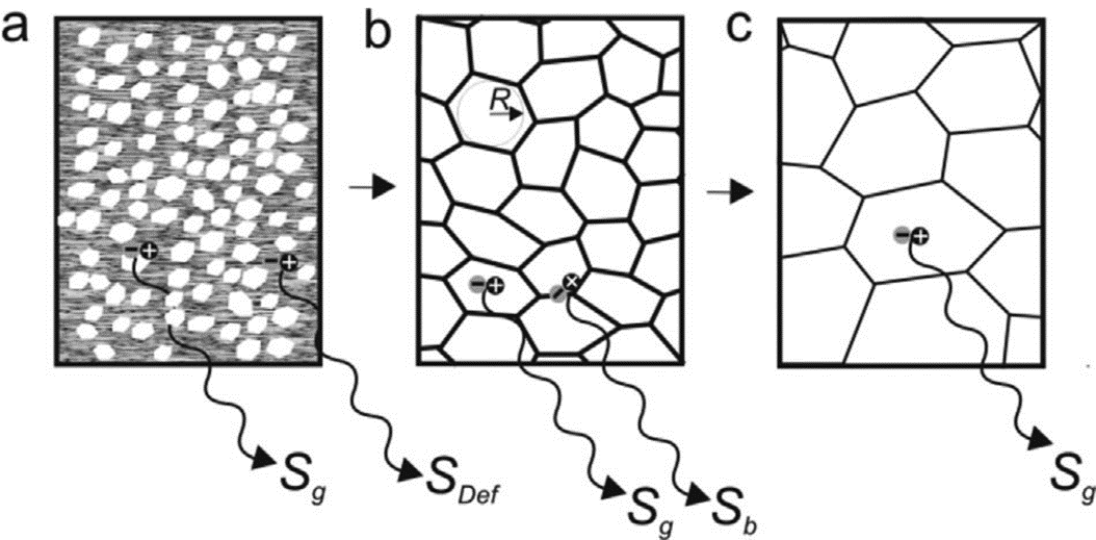
Non-defected

**Figure 11: The measured S-parameter on the thickness reduction of the Ti sample.**

# RESULTS AND DISCUSSIONS

## RECOVERY AND RECRYSTALLIZATION

- Are changes in microstructures of an atom during annealing temperature.



- $S = S_g + (S_b - S_g) \frac{L_+}{R} \left[ \coth \left( \frac{R}{L_+} \right) \right] - \frac{L_+}{R},$

- $\tau = \tau_g + (\tau_b - \tau_g) \frac{L_+}{R} \left[ \coth \left( \frac{R}{L_+} \right) \right] - \frac{L_+}{R},$

- Where  $S_b$  and  $S_g$ - S-parameter.

- $R^m = R_0^m + t \exp \left( -\frac{Q_m}{kT} \right),$

- Where  $R_0$  – initial grain radius;

- $R$  – mean radius;

- $m$  – grain growth exponent;

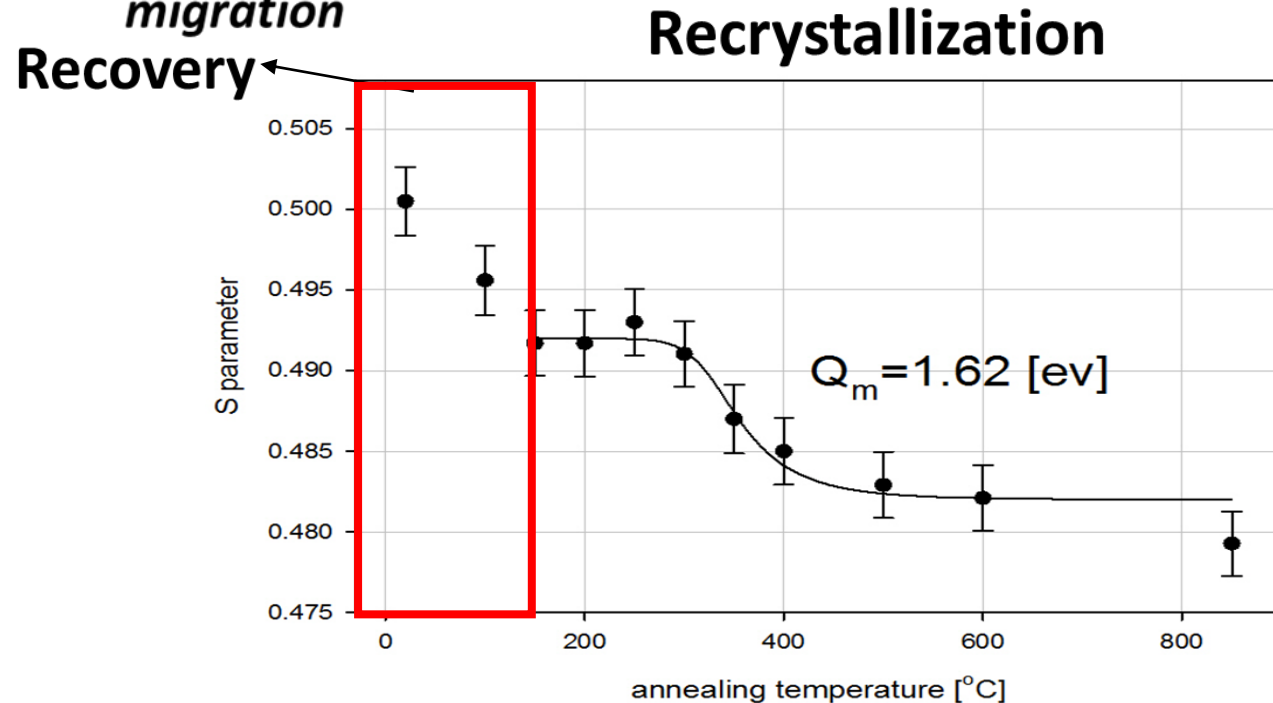
- $t$  – time.

**Figure 12: Schematic diagram of the main processes in the deformed sample exposed to annealing.**

# Doppler broadening and positron lifetime Spectroscopy

- The **S-Parameter** and **Positron lifetime** decreases with an increasing temperature.
- Type of Defects: **Vacancies on dislocations lines**

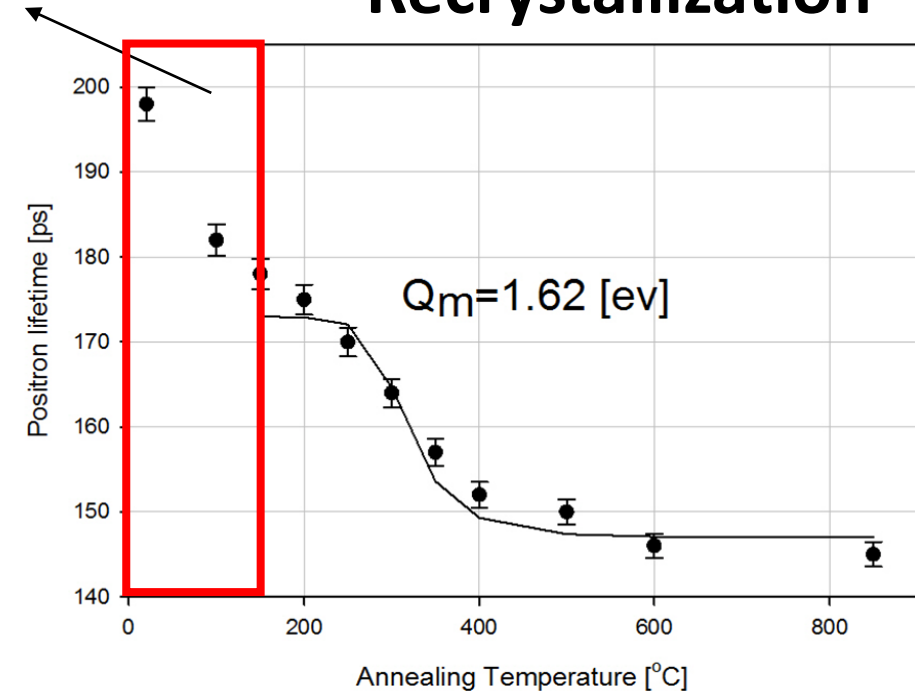
$Q_m$  – Activation energy of boundary migration



**Figure 13: The S-parameter dependency on the annealing temperature of deformed Ti.**

**Recovery**

**Recrystallization**



**Figure 14: Positron lifetime dependency on annealing temperature.**



# CONCLUSION

The aim was to study the **Recovery** and **Recrystallization** on **pure Titanium**. According to obtained results of the positron lifetime, it tells that the type of defects formed are **Vacancies on the dislocation line**. From the dependencies of the positron lifetime and the S-parameter, activation energy of boundary migration  $Q_m$  was determined and equal to **1.62 eV**.

# ACKNOWLEDGEMENTS



*Thank  
you!*