

# Influence of previous heat treatment on the AISI 420 steel low temperature nitriding kinetics

Thiago F. Amaral, Fernando I. Zanetti,  
Cristiano J. Scheuer, Silvio F. Brunatto,  
Rodrigo P. Cardoso

**X** Encontro da  
**SBPMat**

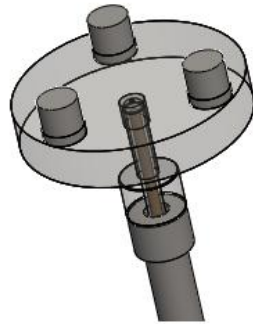
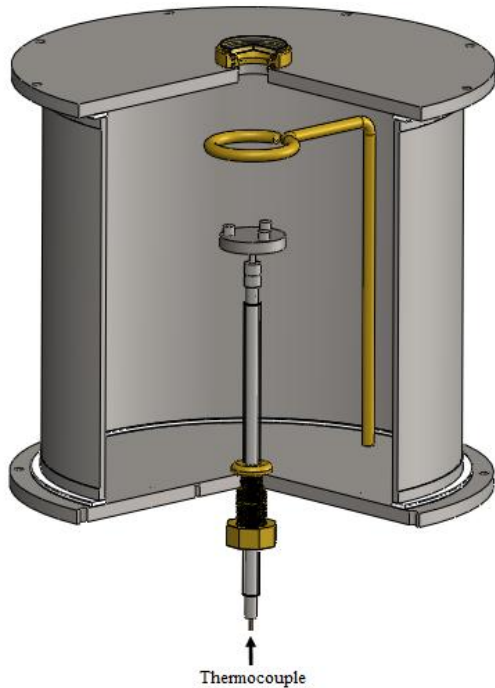
*Gramado-RS*

25 a 29 | setembro  
2011

# Motivation

- Diffusion and precipitation process affects the nitriding process.
- To study the influence of previous heat treatment on the kinetics of low temperature nitriding of the AISI420 martensitic stainless steel.
  - Layer growth kinetics
  - Chromium nitride precipitation that causes sensitization (to be avoided)

# Experimental apparatus



## Fixed parameters:

- Pulse frequency: 4.2 kHz
- Peak voltage: 600V
- Gas mixture: 10%Ar+20% H<sub>2</sub>+70% N<sub>2</sub>
- Gas flow rate: 200 sccm
- Pressure: 3 Torr

## Samples:

As-quenched (air quenched from 1050 °C)  
Tempered (1h 400 °C)  
Annealed (as received) -> **lower chromium**

## Treatment conditions:

- Temperature: 300, 350, 400, 450 and 500 °C for 4h
- Time: 2, 4, 6 and 12 h at 350, 400 and 450 °C

**Characterization:** microstructural analysis, microhardness test and XRD.

# Microstructural analysis

(4h treatment)

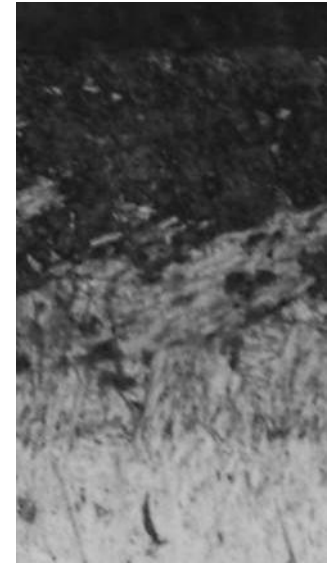
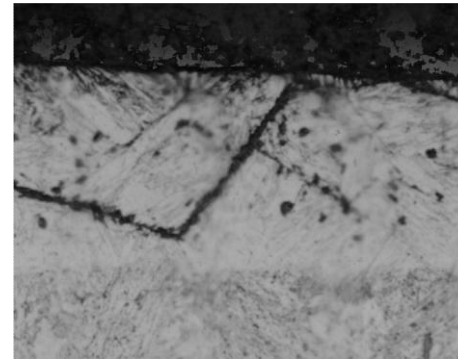
350 °C

400 °C

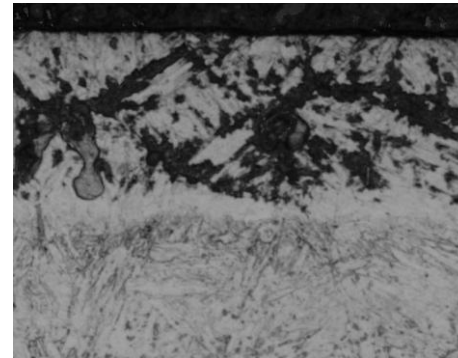
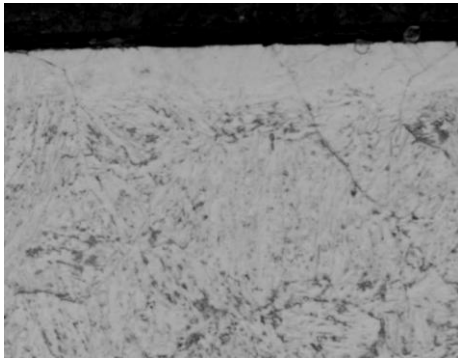
450 °C

500 °C

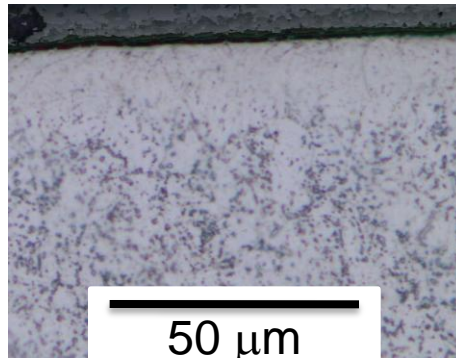
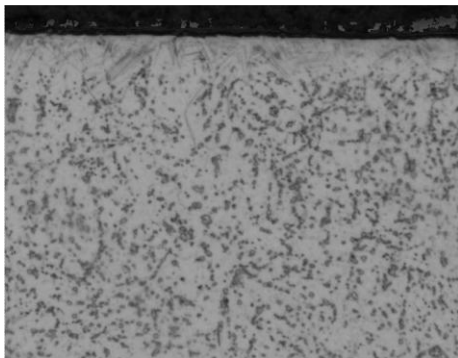
As-quenched



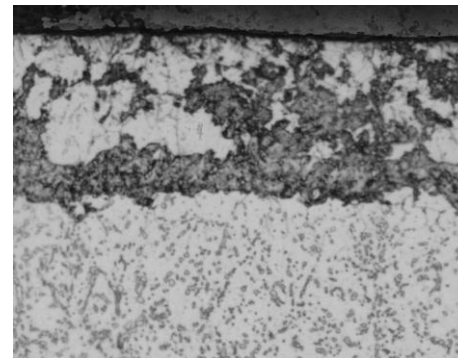
Tempered



Annealed



50 μm



↓ T → ↓ etching  
↑ T → ↑ sens.  
As-quenched → less sens.



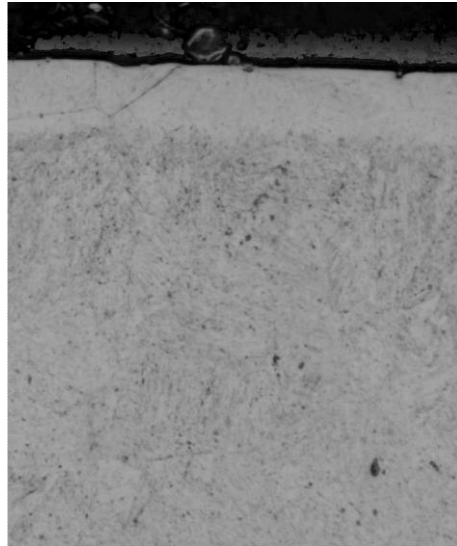
# Microstructural analysis

- Time evolution -> As-quenched samples treated at 400 °C

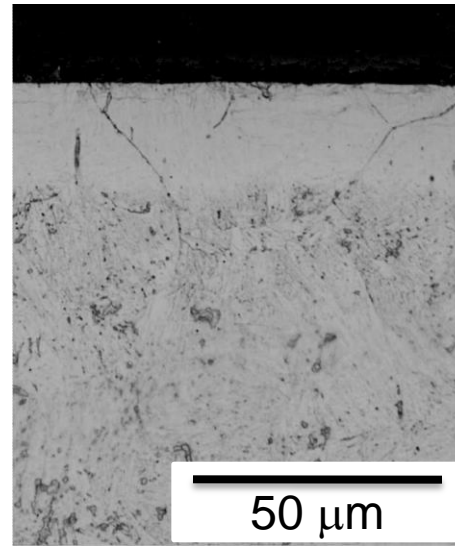
2h



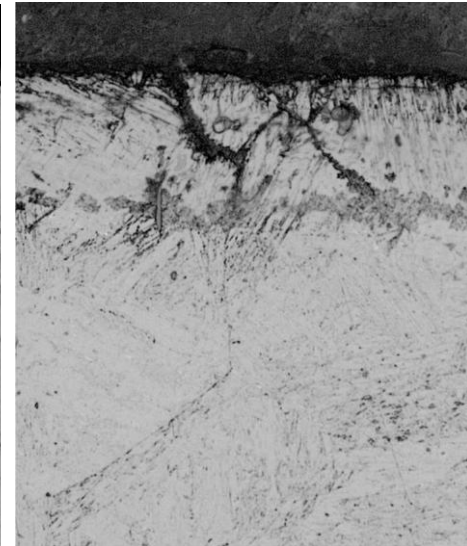
4h



6h



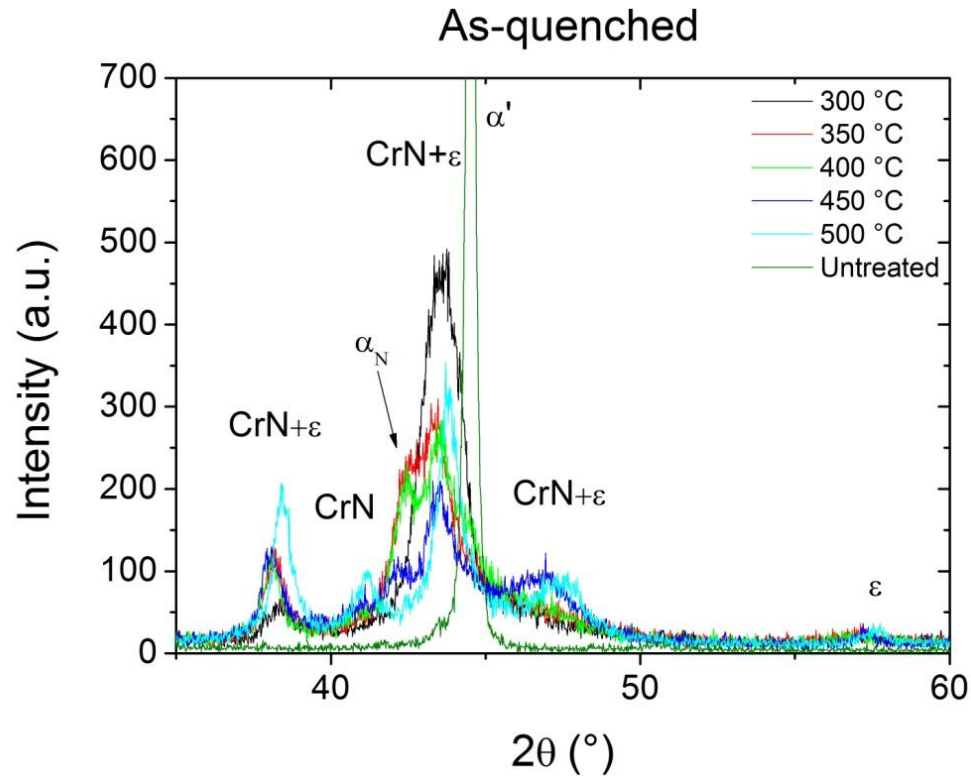
12h



- The sensitization starts at the grain boundary for 4h
- The sensitization is generalized for 12h

# XRD Patterns

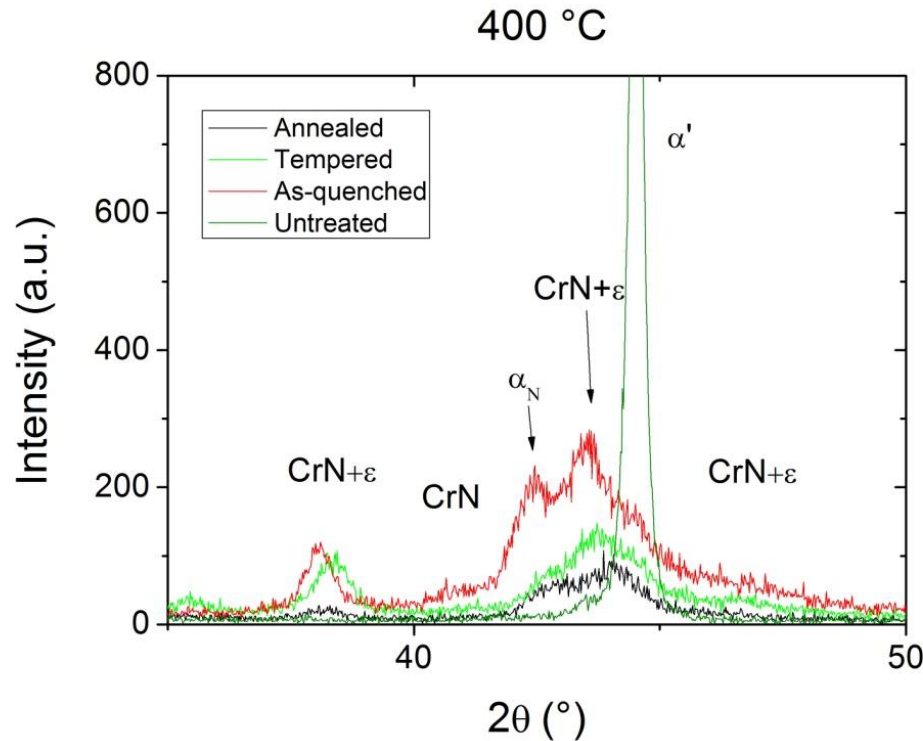
## (Temperature dependence)



- For low treatment temperature the main phases are  $\varepsilon$  and expanded martensite
- For high treatment temperature peaks of CrN are also observed (sensitization)
- The peak of expanded martensite disappears for high temperature, what is expected for a metastable phase

# XRD Patterns

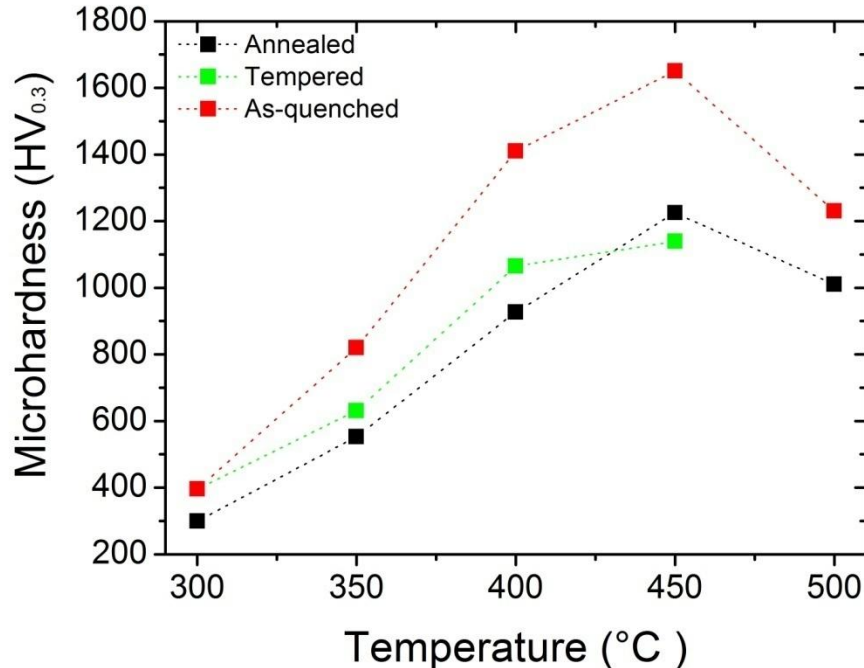
(Influence of the previous heat treatment)



➤ The as-quenched samples present a more intense peak of expanded martensite → higher solid solution chromium content → higher nitrogen supersaturation

# Microhardness

(Temperature dependence – 4h treatment)

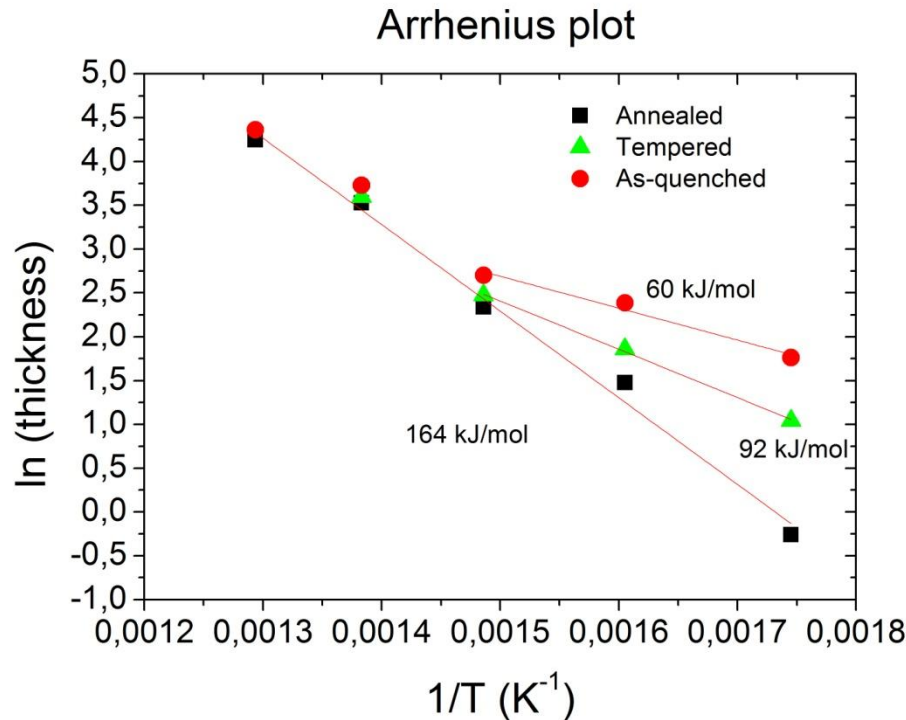


- The effective hardness grows with the temperature up to 450 °C
- The effective hardness is always higher for as-quenched samples
- After chromium nitride precipitation, intense at 450 °C, the effective hardness decreases, indicating that expanded martensite has important hardening effect



# Layer thickness evolution

## (Temperature dependence)



$$\ln(d) = ct. - \frac{Q}{2R} \frac{1}{T}$$

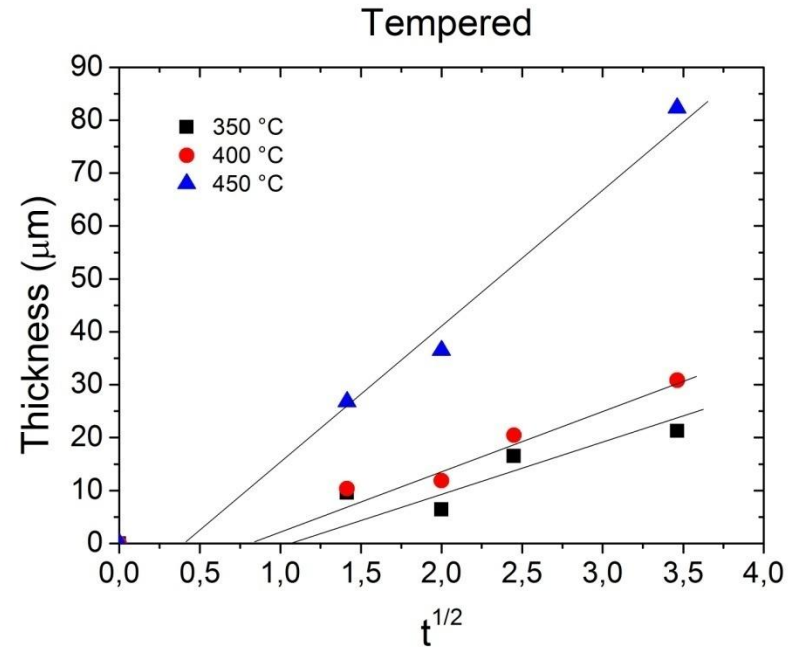
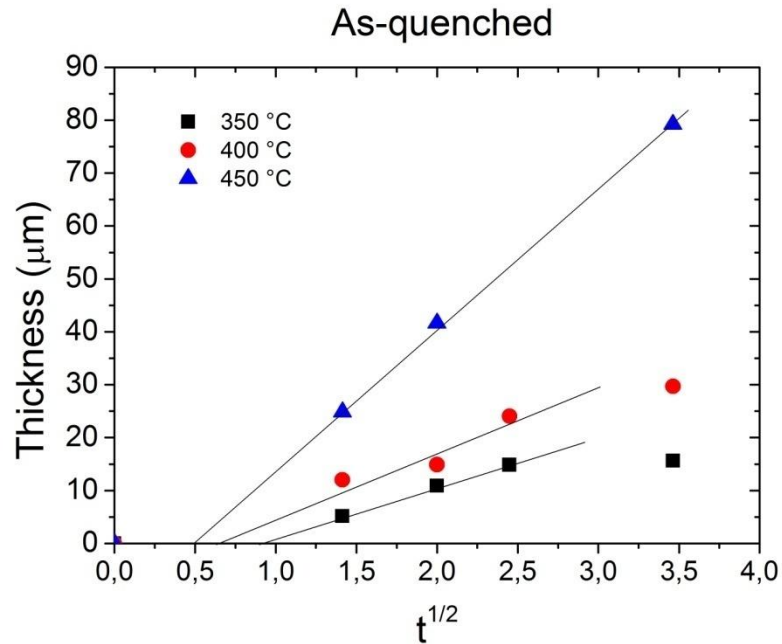
Arrhenius equation  
Linearization considering:

$$d \propto \sqrt{Dt}$$

- At high temperature the limiting process for the layer growth tends to have the same activation energy (tending to thermodynamic equilibrium)
- Activation energy for annealed samples do not change (equilibrium)
- At low temperature the activation energy is lower for as-quenched and tempered samples when compared to the annealed samples (defects density, residual stress and/or chromium content in solid solution)

# Layer thickness evolution

## (Time dependence)



- The incubation time for nitride layer formation is higher for lower treatment temperature
- For low temperatures, precipitation occurs for high treatment time changing the layer growth kinetics
- The temper state do not play an important role on the layer thickness time behavior for the studied condition

# Closing remarks

- The pretreatment dramatically affects the low temperature nitriding of martensitic stainless steel
  - The best results have been obtained for as-quenched samples (simultaneous tempering and nitriding)
- The chromium content in solid solution and/or residual stress plays an important role on the expanded martensite formation

**THANK YOU!**