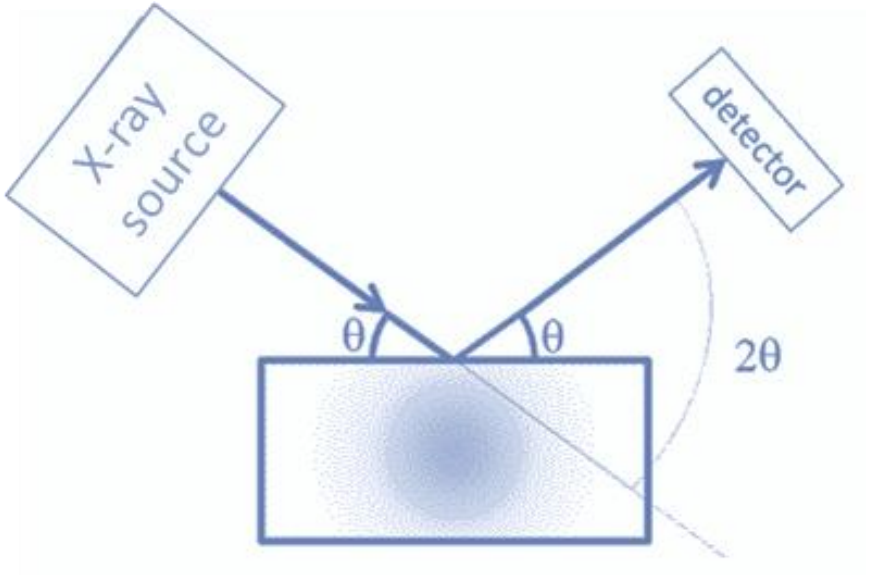
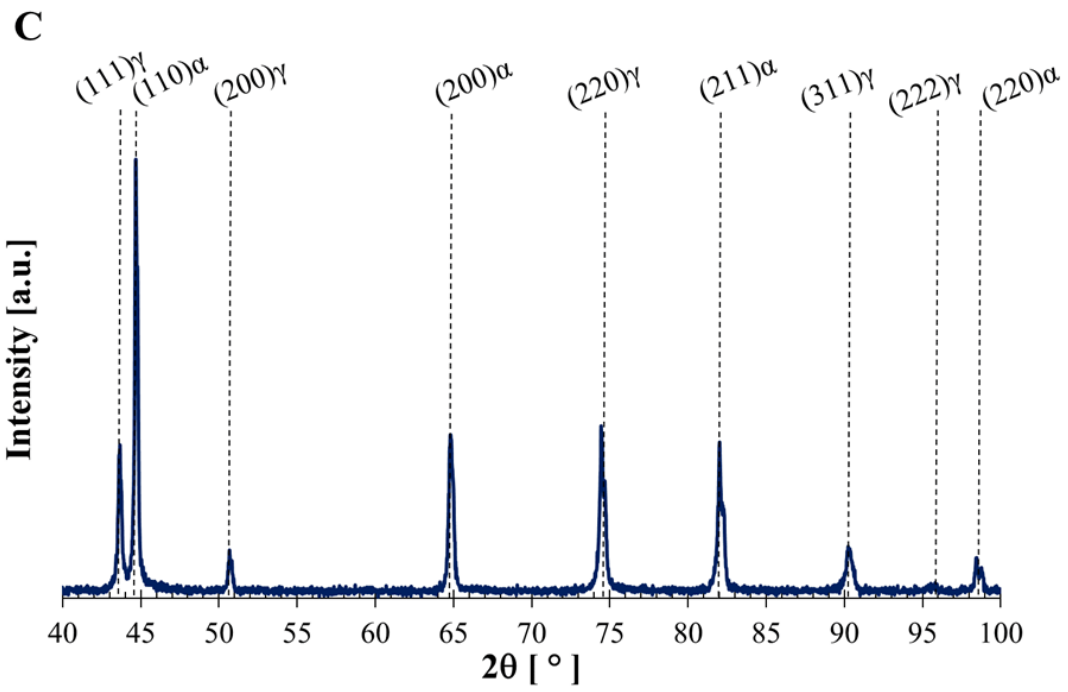
APPLICATIONS OF X-RAY DIFFRACTOMETRY ON PHASE QUANTIFICATION IN STAINLESS STEELS

F. R. Pucci, T. A. Lopes, J. W. Calderon-Hernandez,
RODRIGO MAGNABOSCO

Introduction

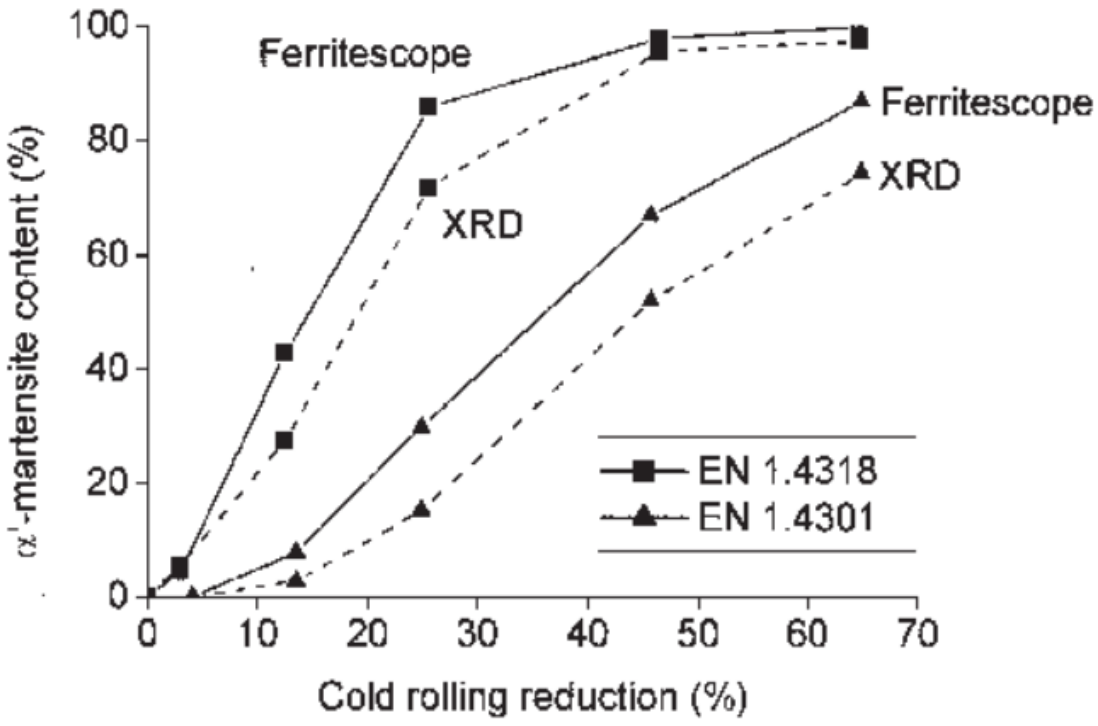
$$d_{(hkl)} = \frac{n\lambda}{2sin\,\theta}$$



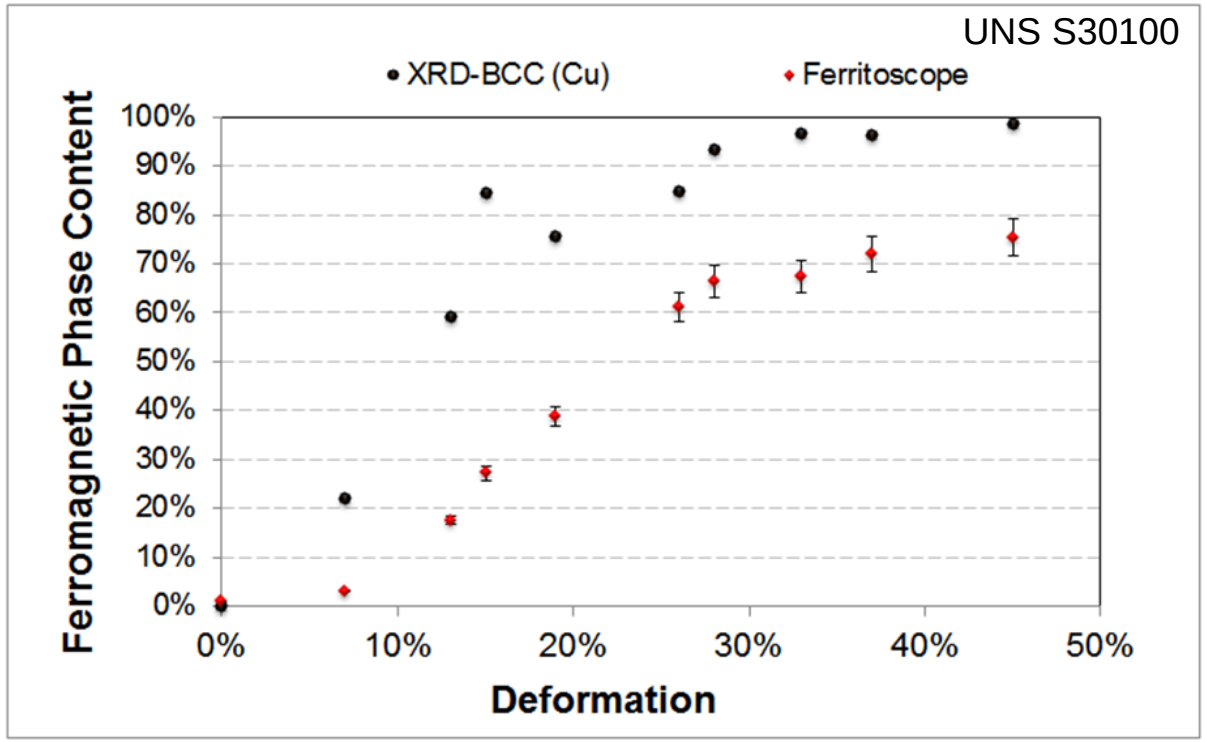


$$X_{\alpha'} = \frac{\frac{1}{n}\sum_1^n \frac{I_{\alpha'}}{R_{\alpha'}}}{\frac{1}{n}\sum_1^n \frac{I_{\alpha'}}{R_{\alpha'}} + \frac{1}{m}\sum_1^m \frac{I_{\gamma}}{R_{\gamma}}}$$

Introduction



J. TALONEN, H. HÄNNINEN, Damping Properties of Austenitic Stainless Steels Containig Strain-Induced Martensite, Mettalurgical and Materials Transactions A, v. 35A, ago. 2004, p. 2401.



J.W. FEREZIN, R. MAGNABOSCO, Study of strain induced martensitic transformation in two austenitic stainless steels by x-ray diffraction. 9th ESSC and 5th European DSS Conference proceedings. 2017. p. 1-7

Objectives

This work evaluates XRD as a technique for phases quantification, compared to ferritscope magnetic measurements.

ferrite in DSS

martensite ($\gamma_{retained/reversed}$) in SMSS

Experimental procedure

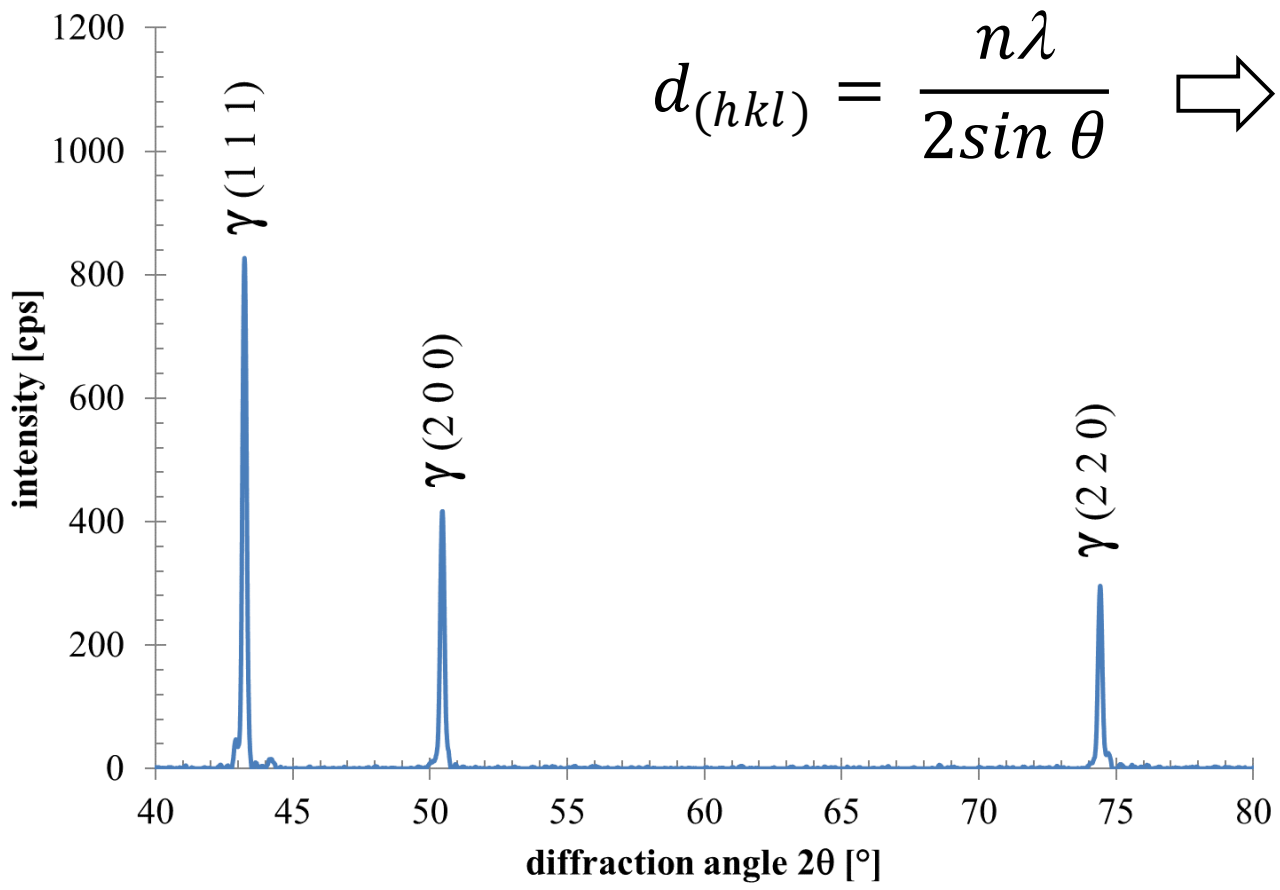
Steel UNS	Cr	Ni	Mo	N	C	Mn	Si	other	Fe
DSS	22.5	5.7	3.2	0.16	0.02	1.4	0.35	0.15 Cu	Balance
SMSS	13.4	5.1	1.1	0.17	0.01	0.63	0.39	0.11 Nb	

- DSS
  - 3 mm sheets, solution-treated 30 min @ 1070, 1130 or 1180 °C, water quenched
- SMSS
  - 10 mm sided square bars, austenitized @ 1050 °C, oil quenched
  - tempered 2 h @ 550, 575, 600, 625, 650 or 700 °C
- Samples were metallographic polished prior to XRD measurements
- Ferritscope: 10 to 30 measurements in each sample

Experimental procedure

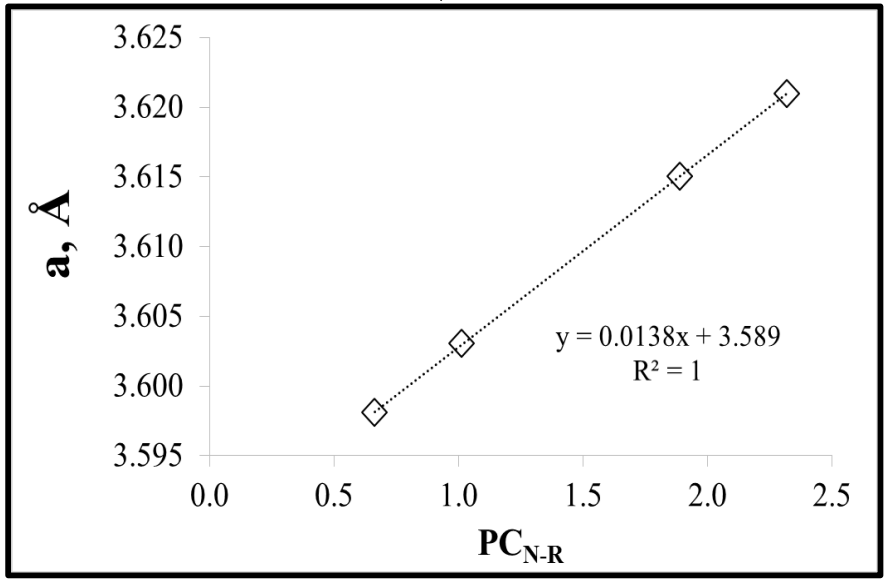
X-ray source	Cu Kα1	Cr Kα1
wavelength (λ, nm)	0.15406	0.22897
scanning angle range	40° < 2θ < 100° for DSS 40° < 2θ < 85° for SMSS	60° < 2θ < 160°
step	0.02 °	
scanning rate	0.5 °/min	
X-ray power	0.9 kW	
scannings per sample	10	

Experimental procedure



$$d_{(hkl)} = \frac{n\lambda}{2sin\,\theta} \Rightarrow a = d_{(hkl)}\sqrt{h^2 + k^2 + l^2}$$

$$CP_{N-R} = \frac{1}{2}\left(\frac{\cos^2\theta}{\sin\theta} + \frac{\cos^2\theta}{\theta}\right)$$



Corrected value: a = 3.589 Å

Experimental procedure

$$R_{hkl} = \left(\frac{1}{V^2}\right)[F^2 p(\frac{1+\cos^2 2\theta}{\sin^2\theta\ \cos\theta})]DWF$$

$$DWF_{(hkl)} = e^{-0,71.\left(\frac{\sin\theta}{\lambda}\right)^2}$$

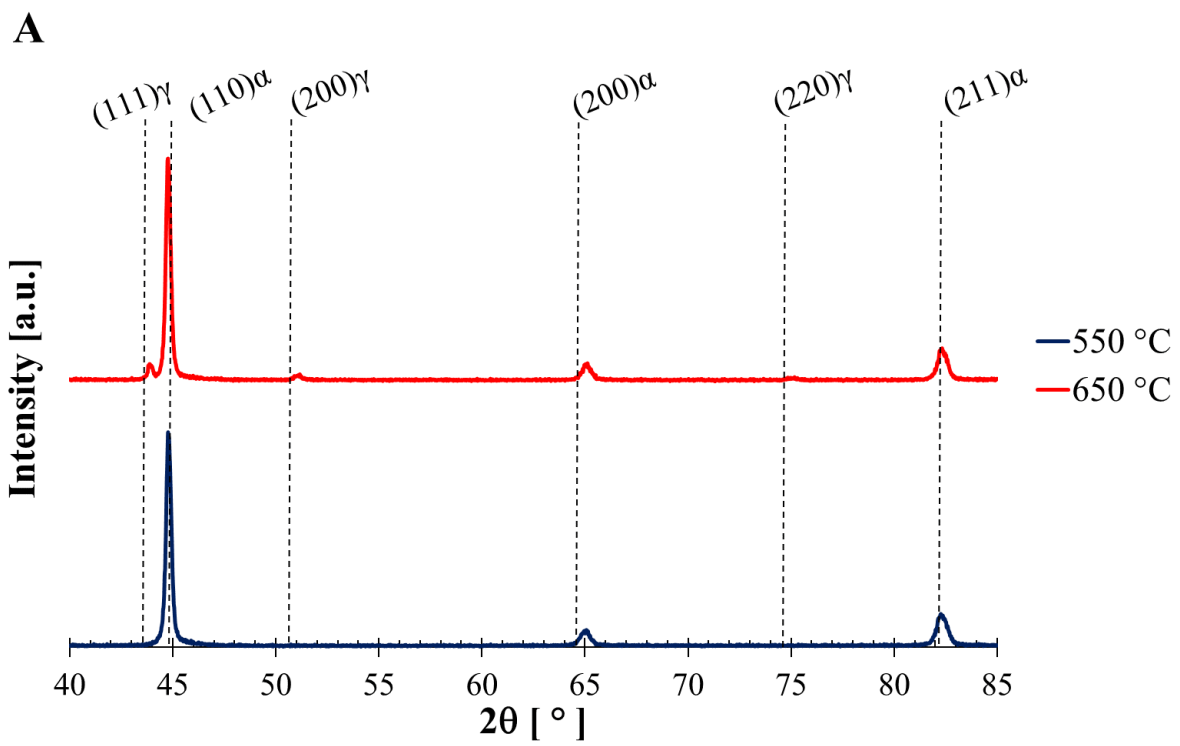
$$V^2 = a^{3^2}$$

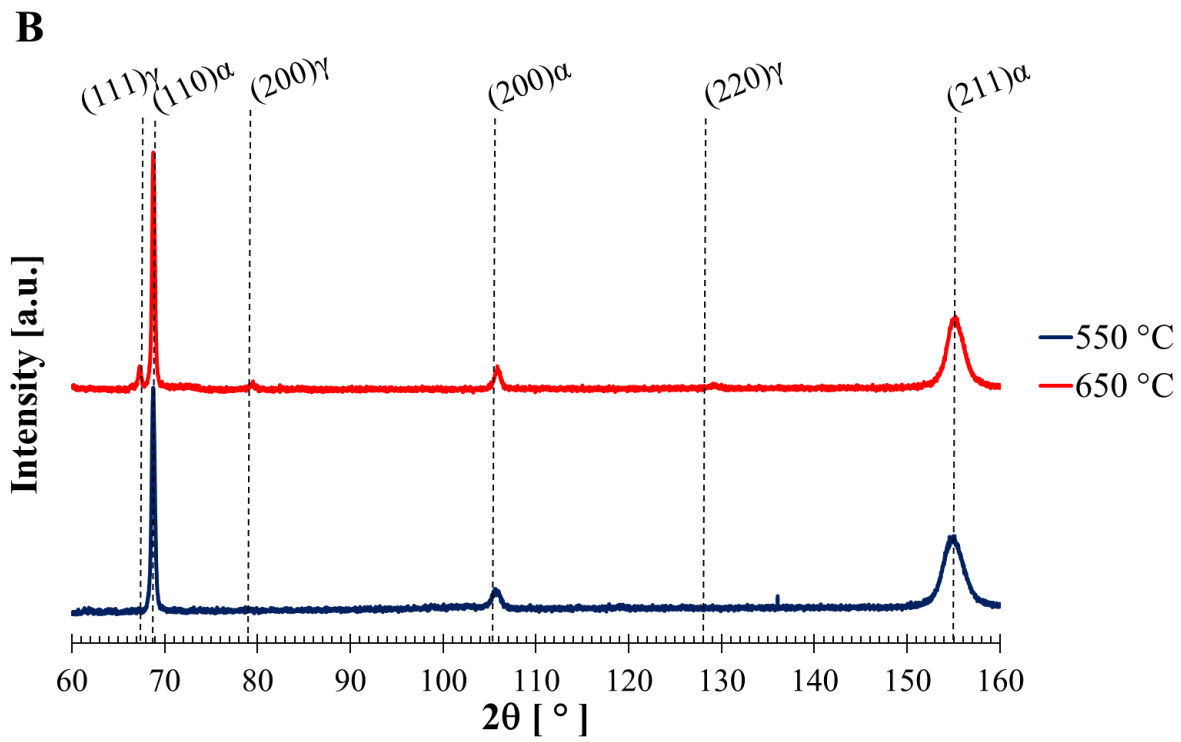
plane	phase	p	F²
(1 1 1)	γ	8	4873
(1 1 0)	α'	12	1197
(2 0 0)	γ	6	4324
(2 0 0)	α'	6	864
(2 2 0)	γ	12	2999
(2 1 1)	α'	24	676

$$X_{\alpha'} = \frac{\frac{1}{n}\sum_1^n \frac{I_{\alpha'}}{R_{\alpha'}}}{\frac{1}{n}\sum_1^n \frac{I_{\alpha'}}{R_{\alpha'}} + \frac{1}{m}\sum_1^m \frac{I_{\gamma}}{R_{\gamma}}}$$

Results and Discussion

SMSS: austenite was only detected at 625 or 650 °C

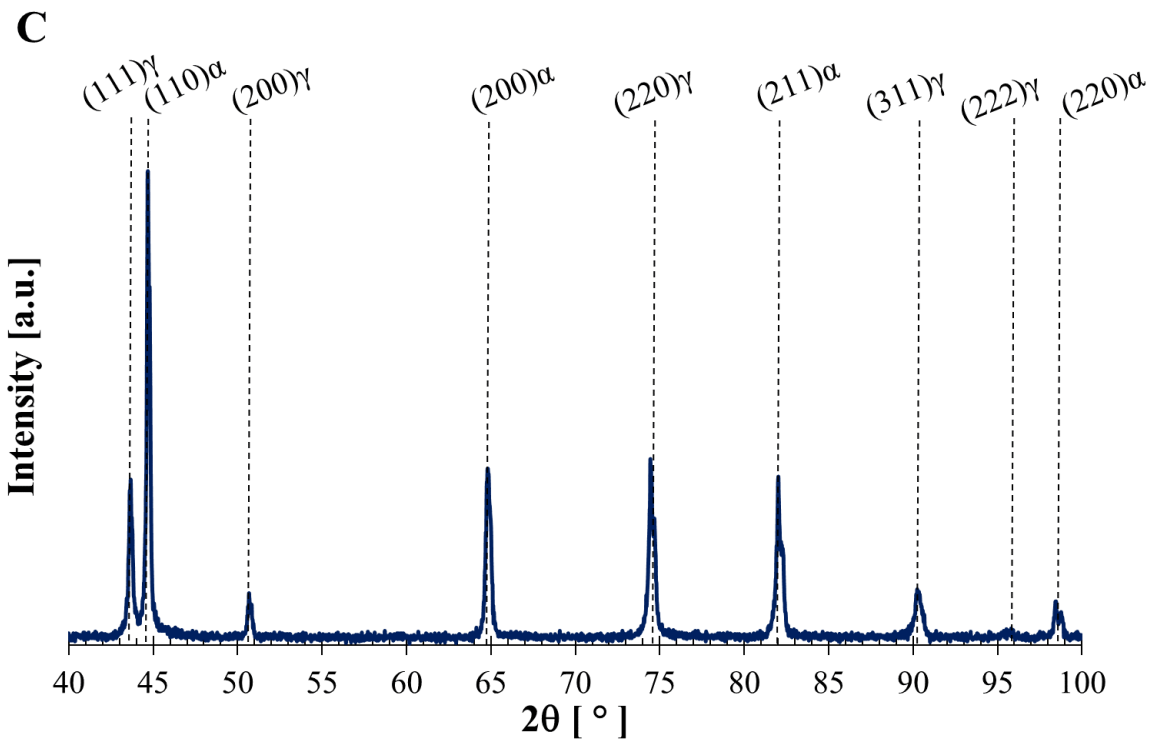


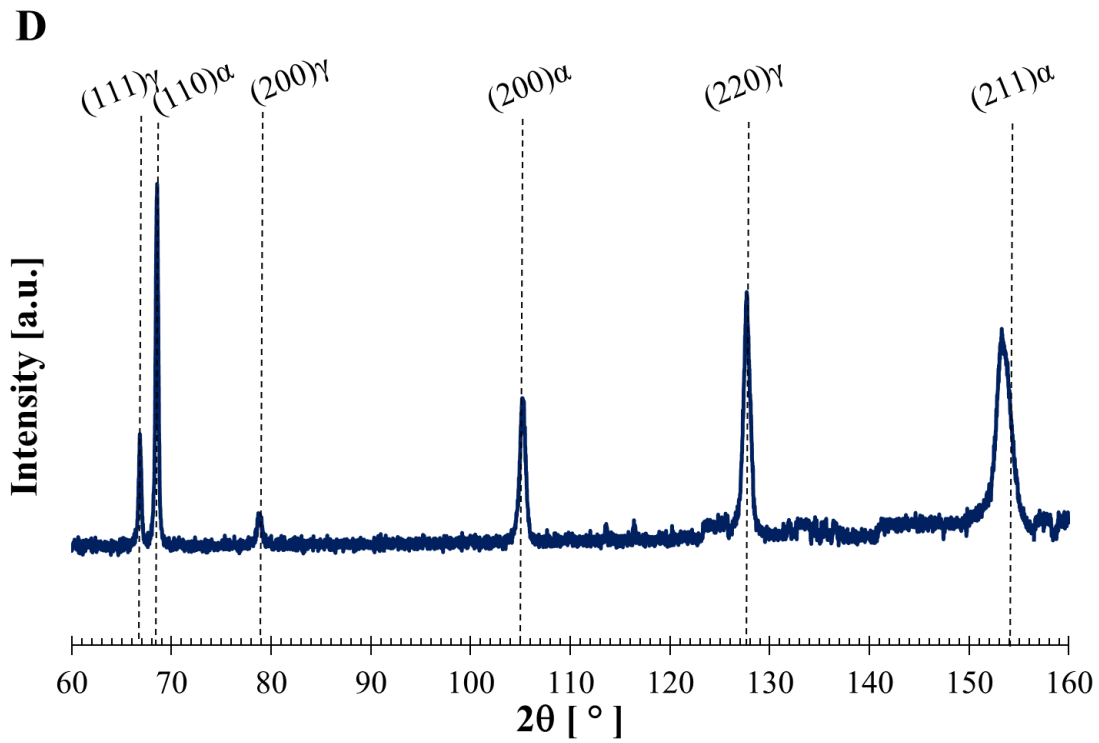


Cu Kα1 | **Cr Kα1**

Results and Discussion

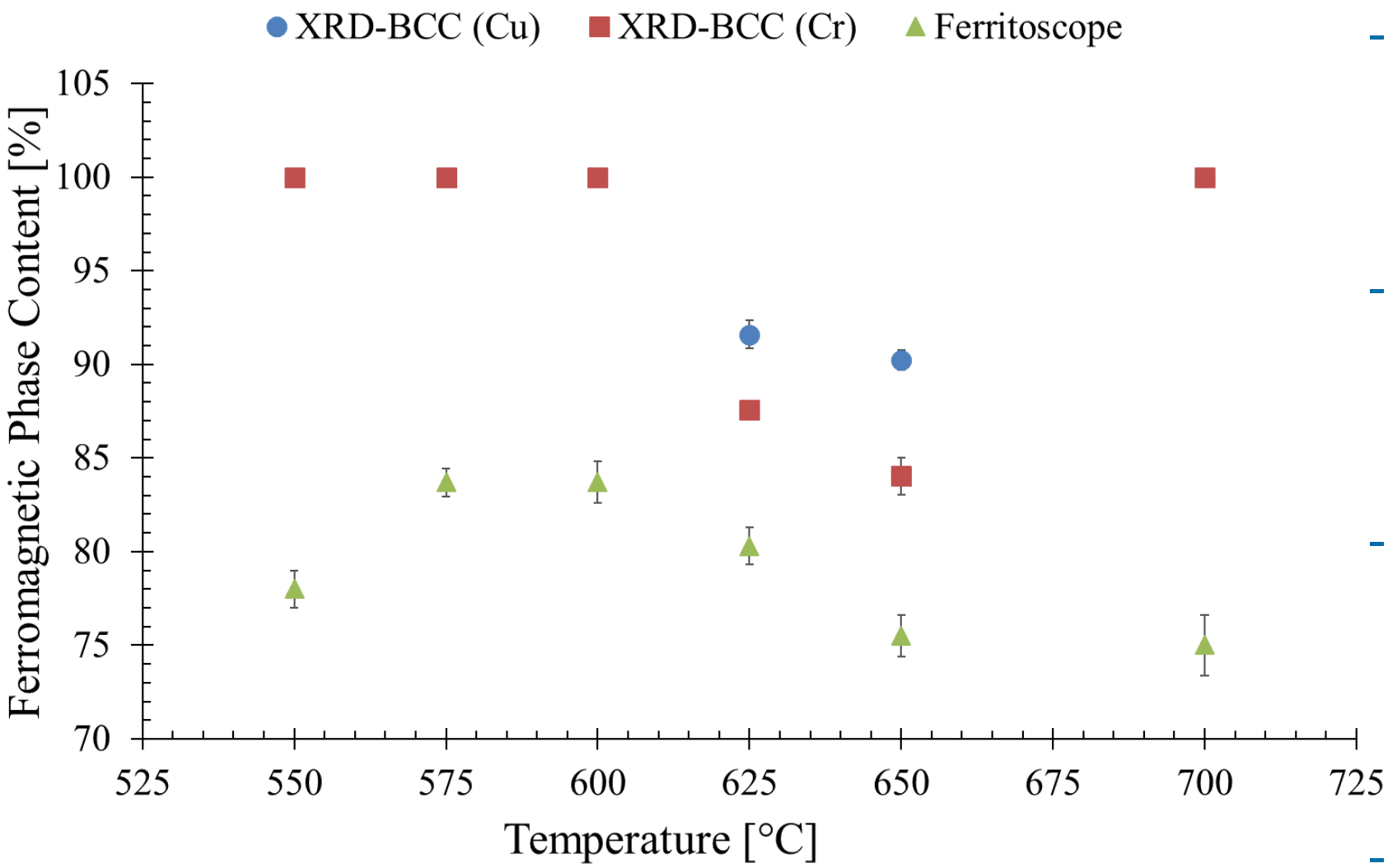
DSS: both phases are detected





Cu Kα1	Cr Kα1

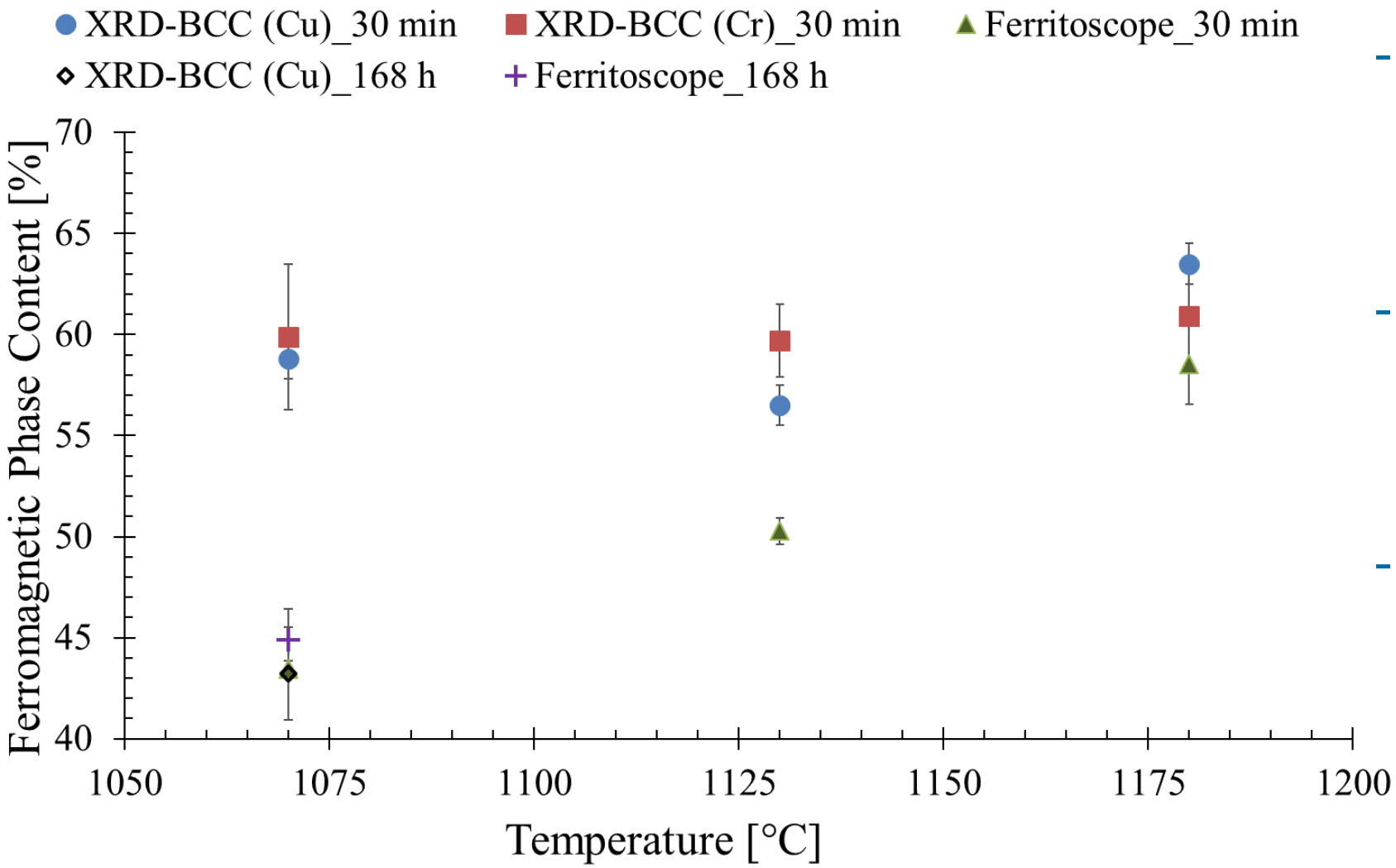
Results and Discussion



SMSS:

- retained (or reversed) austenite was detected in ferritscope at all T.
- small thickness of interlath austenite (specially retained at lower T)
- higher amounts of reversed austenite (700 °C) lead to more martensite formation after tempering
- ***Cr better than Cu***

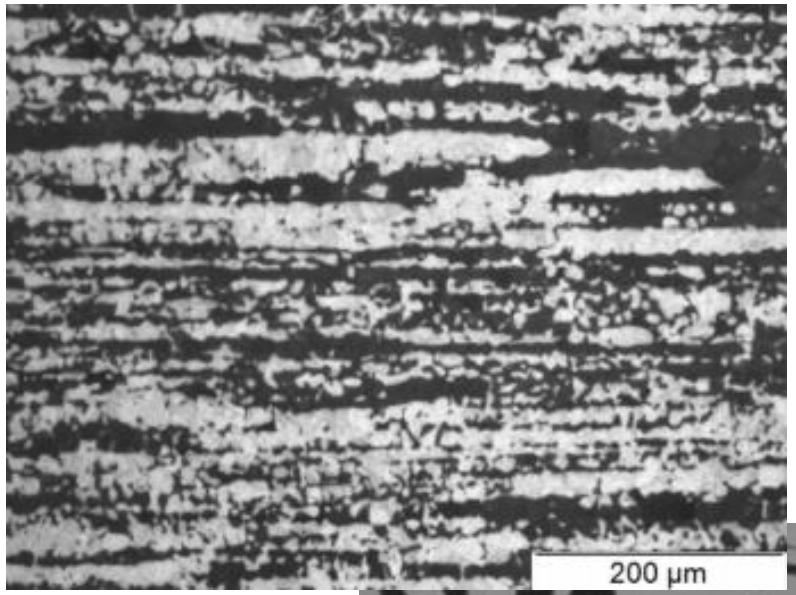
Results and Discussion



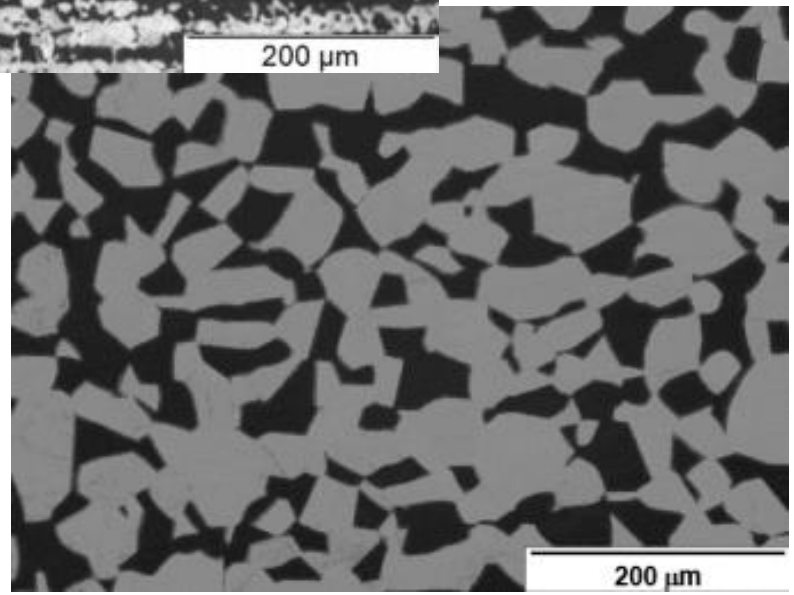
DSS:

- Ferritscope showed smaller values than XRD (confirming ESSC 2017 paper).
- Performance of Cu or Cr radiation seemed to be equivalent
- Lower temperature: huge differences

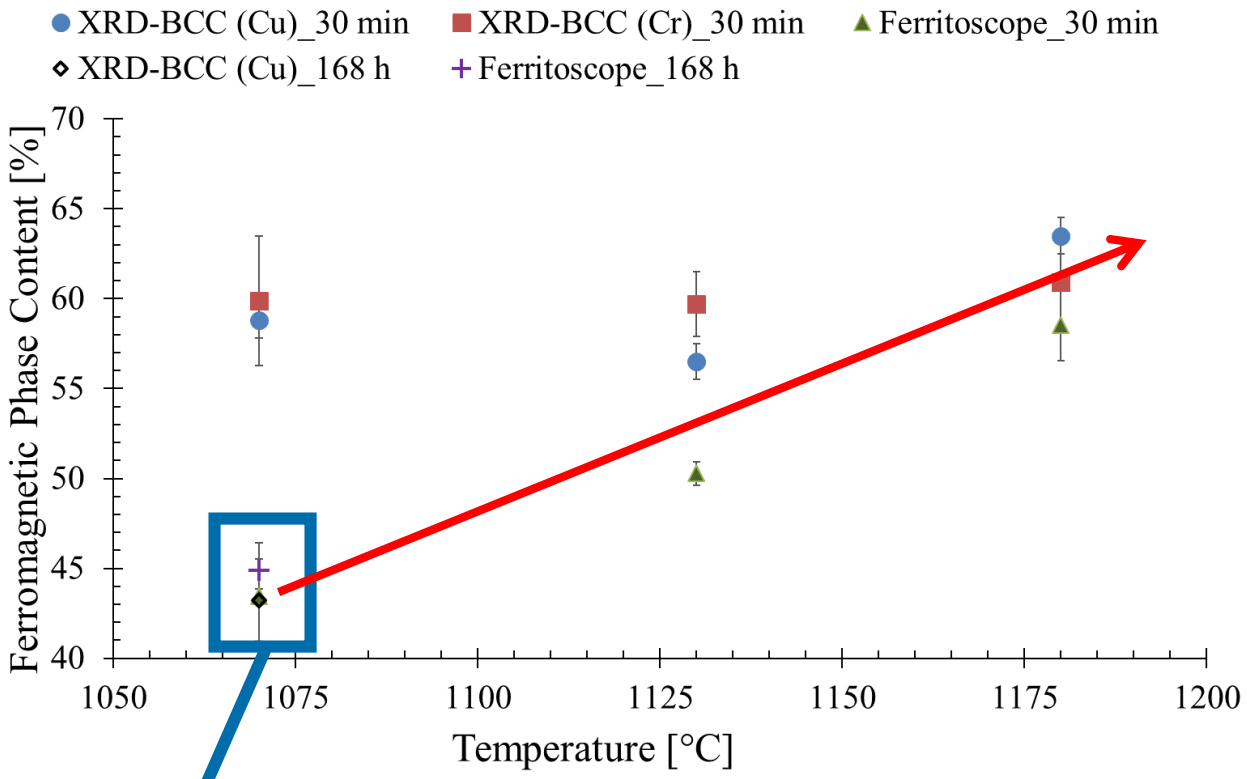
Results and Discussion



30 min
@ 1070°C



168 h
@ 1070°C



Equivalent ferritscope measurements

Higher ferrite content with increasing solution temperature well observed

Conclusions

Ferritscope analysis of SMSS showed that retained and/or reversed austenite is present in all tempering conditions. However, XRD quantification were only able to detect and quantify austenite in samples where the morphology of austenite lead to a higher volume, enhancing austenite peaks intensity.

Conclusions

For the SMSS, the better separation between γ(111) and α(110) peaks when Cr Kα1 radiation was used lead to a better quantification of the reversed austenite formed at 625 °C and 650 °C tempered samples. However, detection of thinner laths of retained austenite was not possible even using Cr Kα1 radiation.

Conclusions

For the DSS, quantification of phases by XRD was only possible using Cu Kα1 radiation, and the samples cannot present preferential orientation of phases.

Acknowledgements

Grant 301458/2016-2

APPLICATIONS OF X-RAY DIFFRACTOMETRY ON PHASE QUANTIFICATION IN STAINLESS STEELS

RODRIGO MAGNABOSCO

rodrmagn@fei.edu.br