Residual stress analysis on oxide layers obtained by oxidation

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- Remarques and perspectives

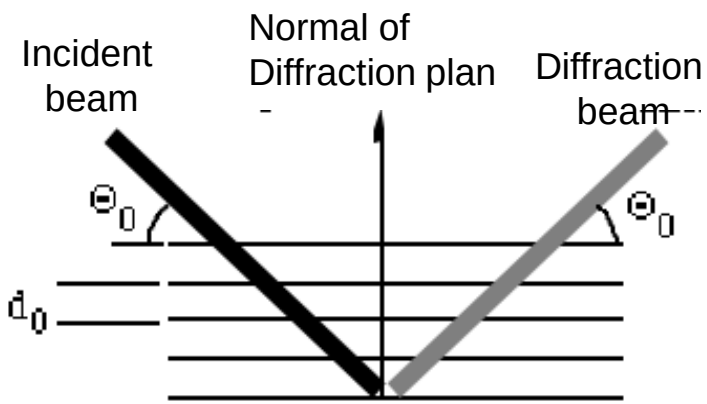
Objectifs

1. Residual stress generated during oxidation (growth stress, chemical gradient etc…) and cooling (thermal stress)

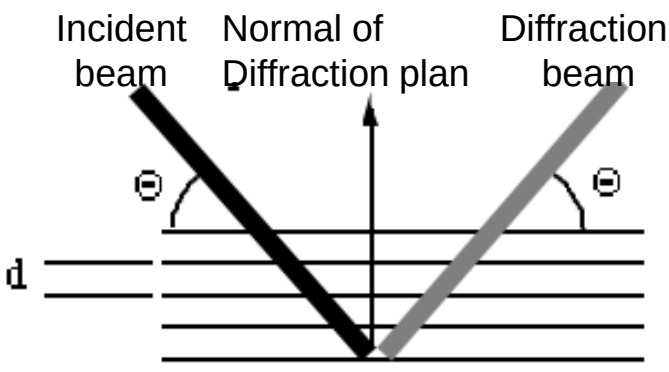
2. Residual stress related to oxidation kinetics, oxide microstructure, durability

3. XRD stress analysis method: non-destructive, selective, surface analysis

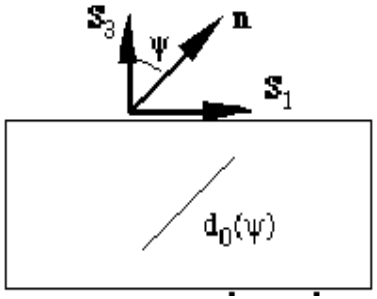
Principle of XRD stress analysis



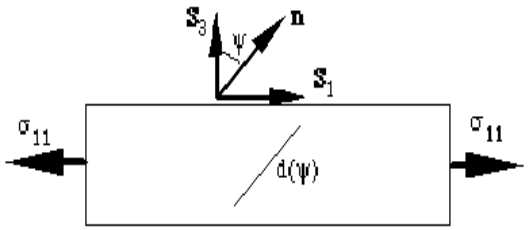
Without deformation



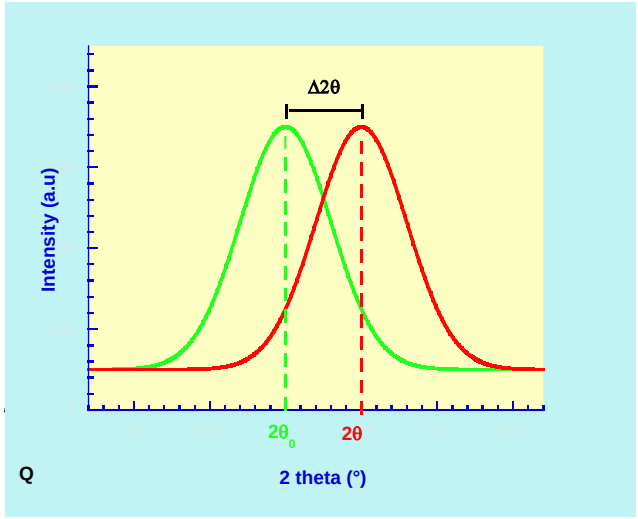
With deformation



Without deformation

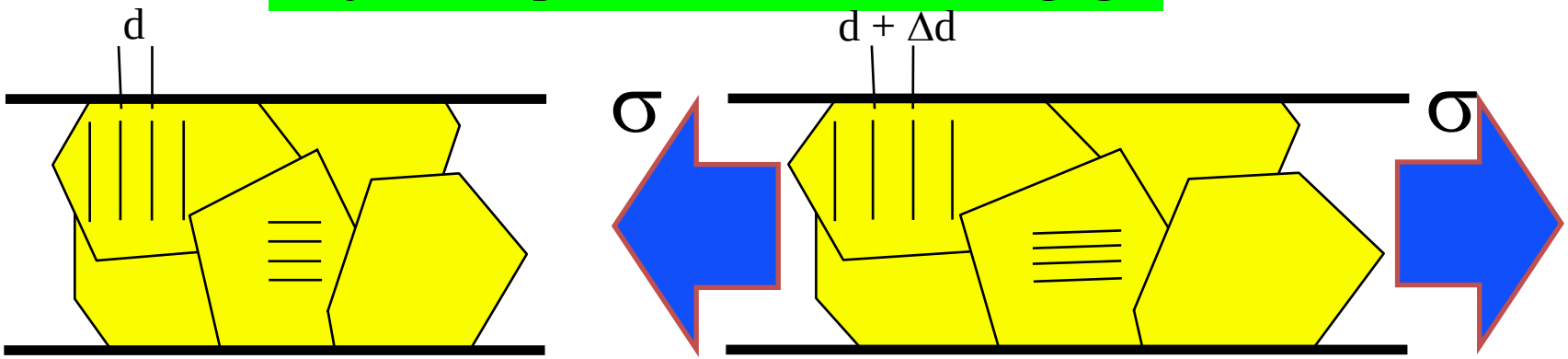


With deformation



Principle of XRD stress analysis

Crystallin plans = Deformation gage



Homogeneous elastic deformations of crystals in material

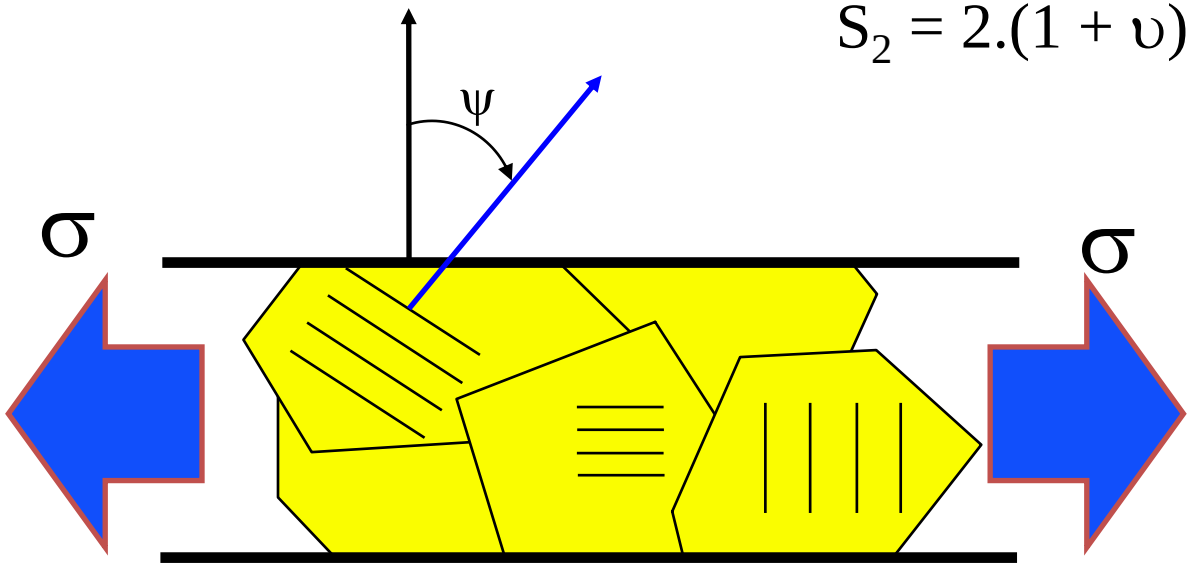
Variations of inter-reticular distances = macroscopic deformations

Principle of XRD stress analysis

$\sin^2\psi$ law:

$$\varepsilon_{\phi\psi} = \frac{1+\nu}{E}\sigma_\phi \sin^2\psi - \frac{\nu}{E}\mathrm{Tr}(\boldsymbol{\sigma}) = \frac{1}{2}S_2.\sigma_\phi.\sin^2\psi + S_1.\mathrm{Tr}(\boldsymbol{\sigma})$$

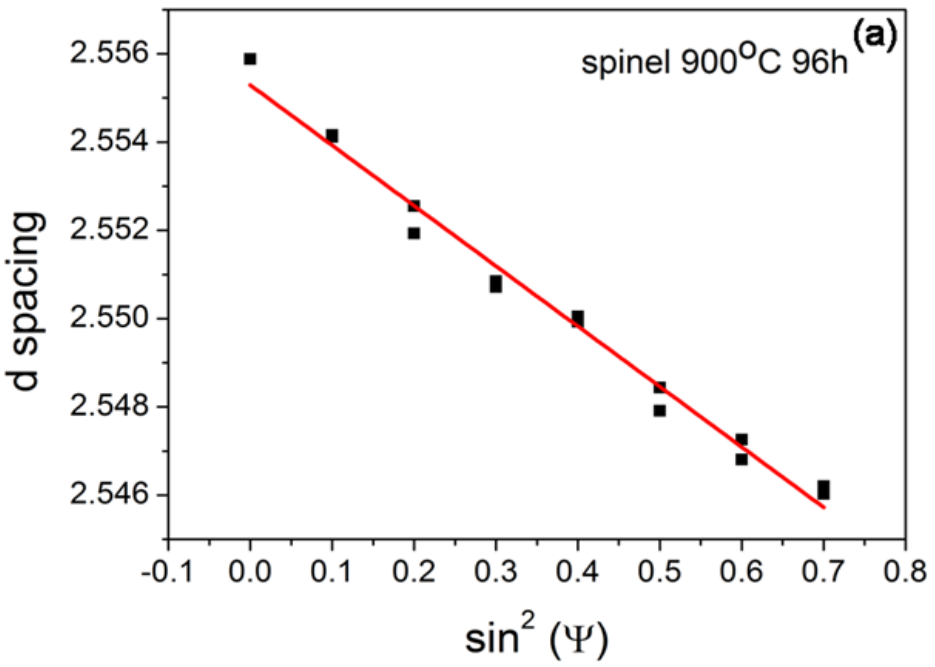
$$S_2 = 2.(1+\upsilon)/E \qquad S_1 = -\upsilon/E$$

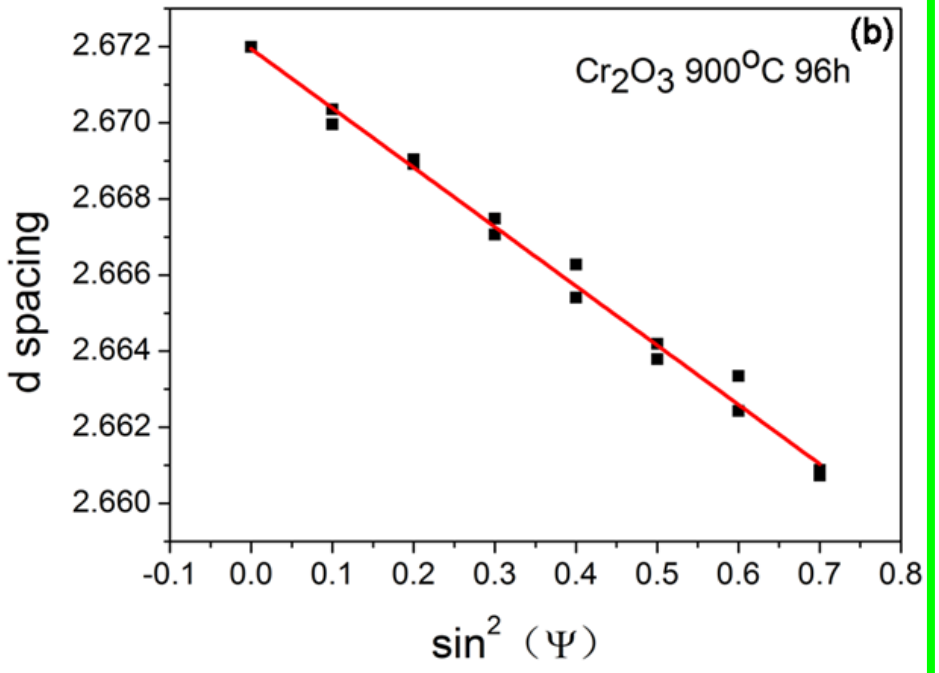


Assumptions

* Homogeneous and isotropical materials
* Elastic strain
* Surface analysis

Principle of XRD stress analysis





Example of $Sin^2\psi$ courves for the two studied phases ({104} for Cr_2O_3 and {311} for $Mn_{1.5}Cr_{1.5}O_4$)

Materials and oxidation conditions

Inconel 600: Ni-17%Cr-8%Fe-1%Mn :

High temperature (600° C-900° C) 6h-150h
Under dry and/or humid air (3%-12% absolute humidity)

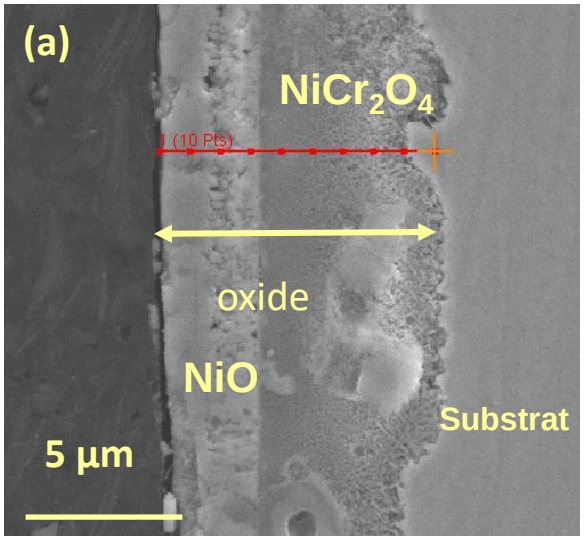
AISI 430 Steel: Fe-17%Cr-1%Mn

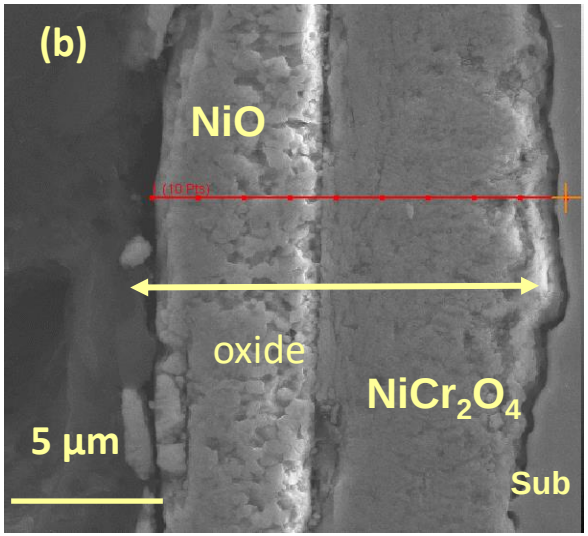
High temperature(700° C-900 ° C), 6h-96h
Under dry air and/or humid air (2%-10% absolute humidity)

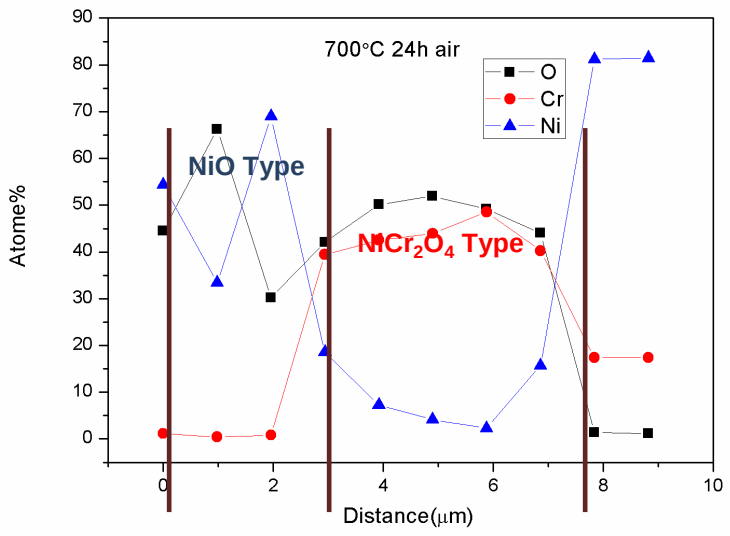
316L: Fe-16%Cr-10%Ni-2%Mo-1%Mn :

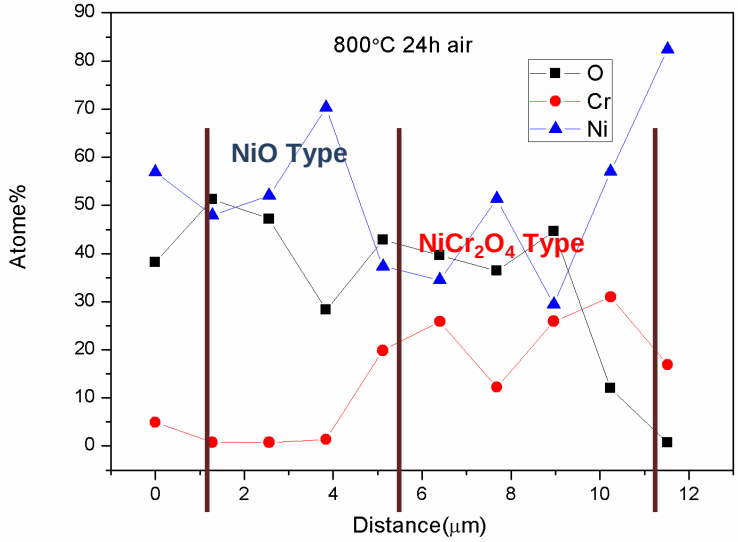
High temperature (700° C-900° C) 2h-96h
Under dry and/or humid air (1%-8% absolute humidity)

FEG-SEM (INCONEL 600)



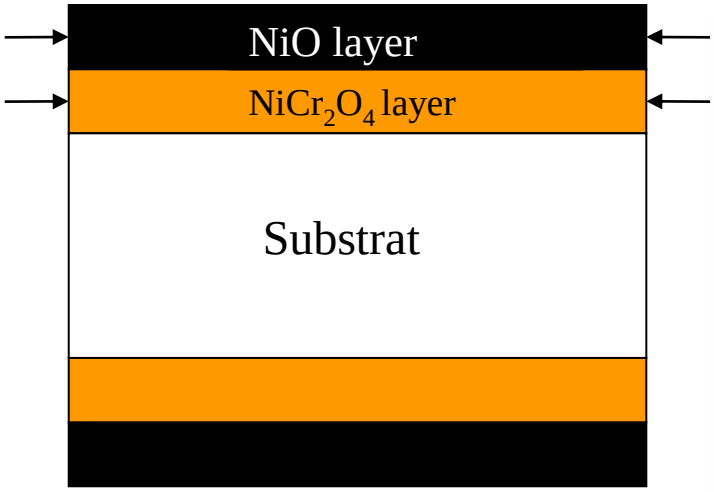


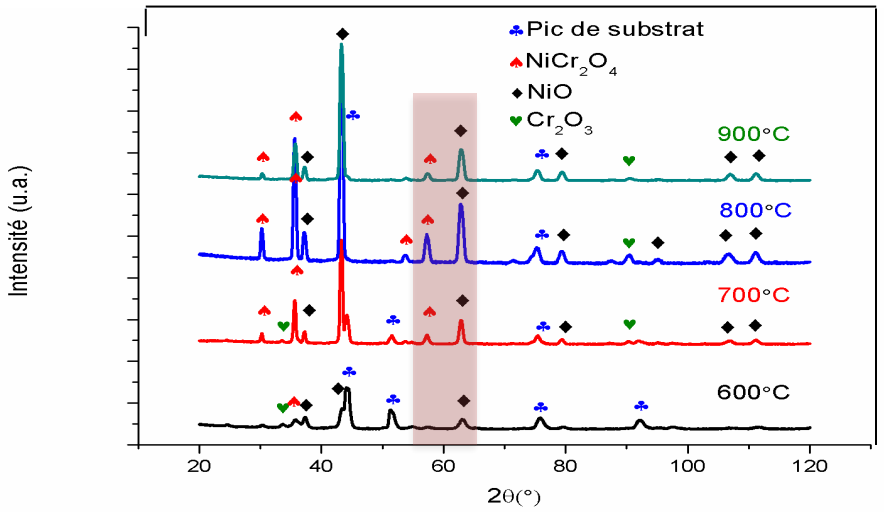




cross-section observation

XRD (INCONEL 600)

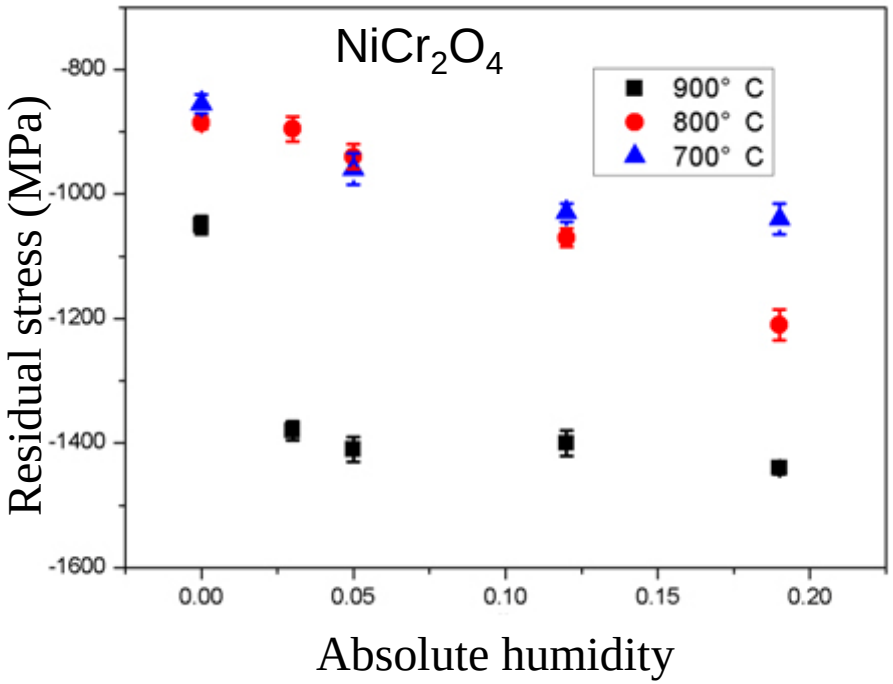


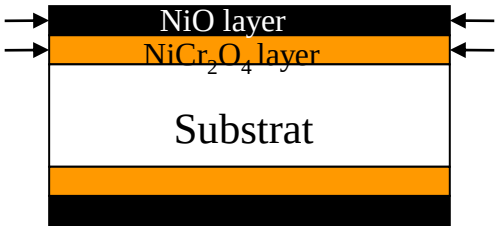


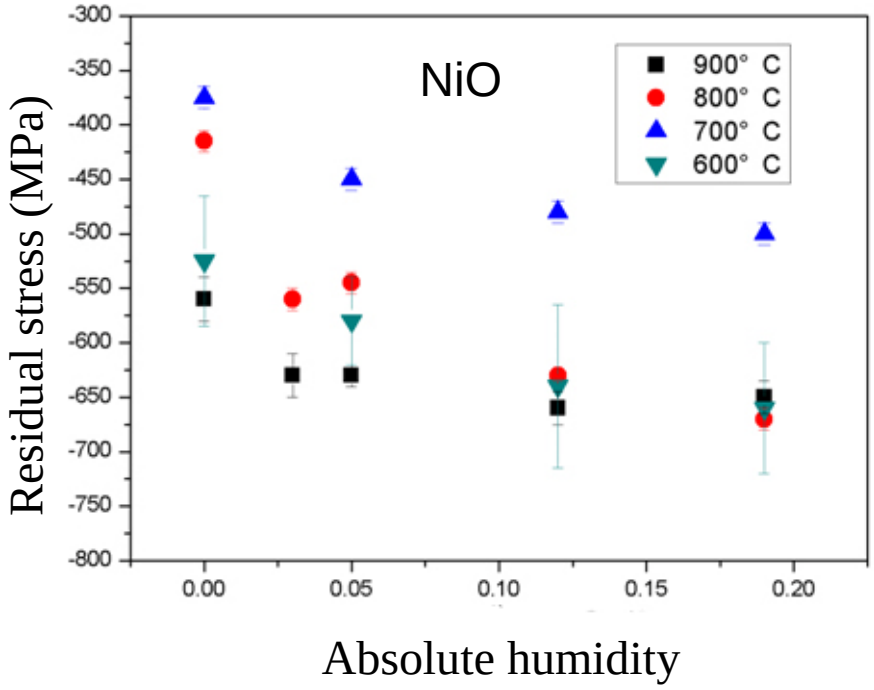
Analysis zone	2mm x 2mm
ψ angle amplitude	[-60° ; 60°]
Number of ψ angle	13
diffraction angle 2θ	63° for NiO . 57° for $NiCr_2O_4$
2θ angle interval	[61.6-64.4]. [55.6-58.4]
2θ step	0.04
Acquisition time	15 s
Precision	30 MPa
Young's modulas	NiO : 190 GPa ; $NiCr_2O_4$: 280 GPa
Poisson's coefficient	NiO :0.25 ; $NiCr_2O_4$: 0.29

Residual stress analysis in two oxide layers

XRD (INCONEL 600)

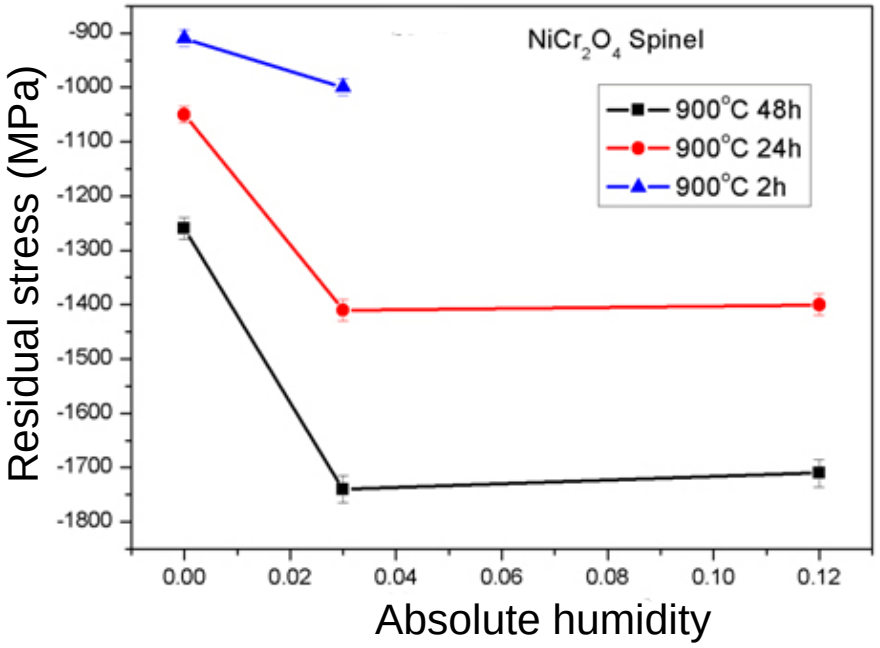


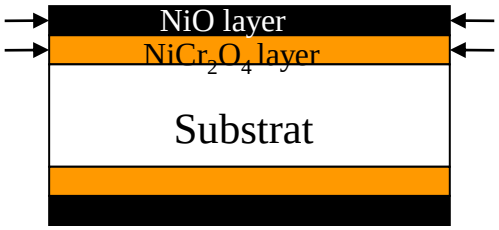


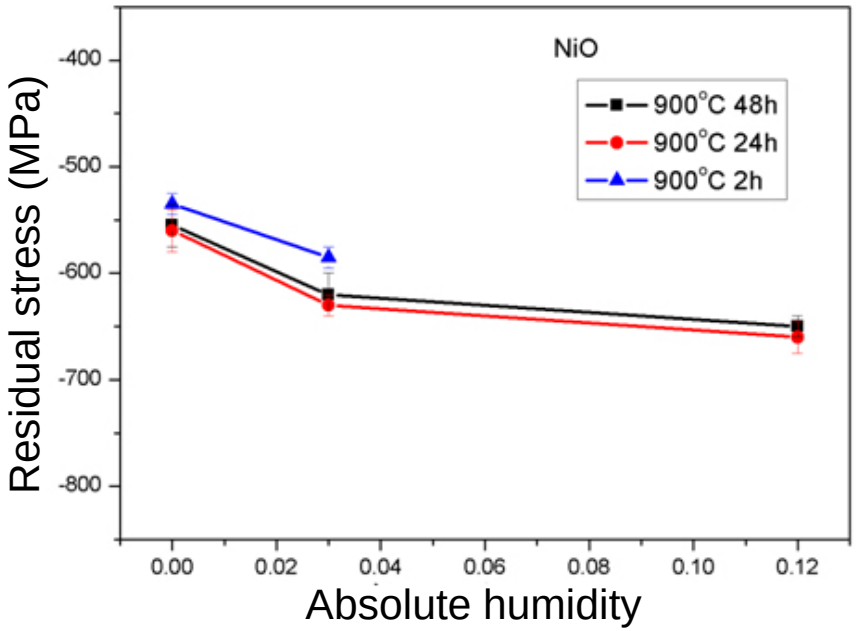


Residual stress analysis
in two oxide layers (after 48h oxidation)

XRD (INCONEL 600)



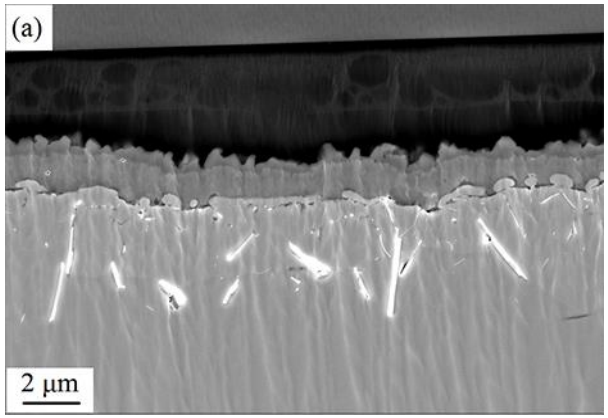


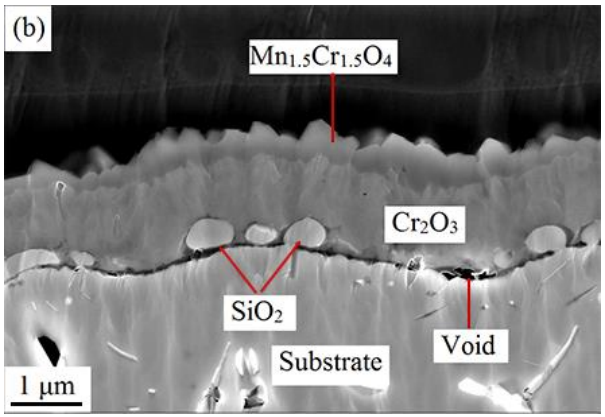


residual stress at 900 ◦ C under various humidy with different oxidation time

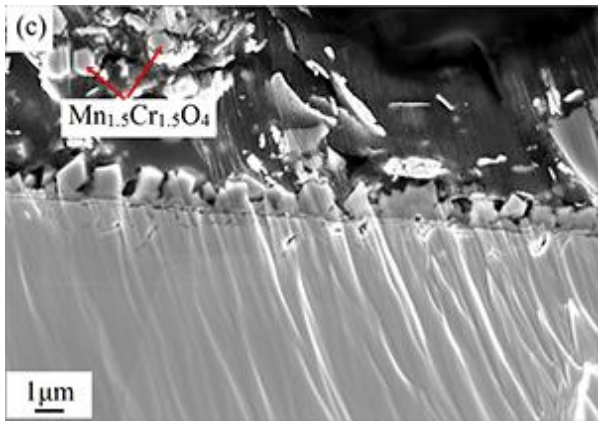
FEG-SEM (AISI430)

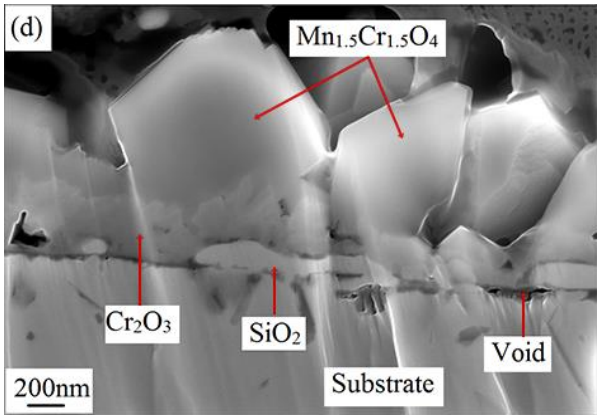
900° C for 12h, 5% AH





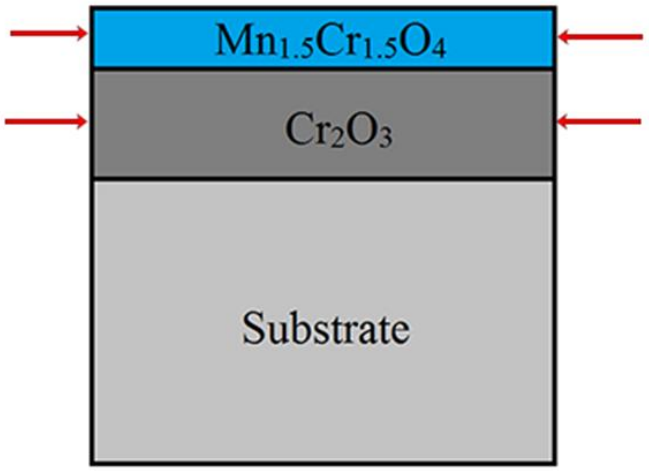
800° C for 48h, 5% AH

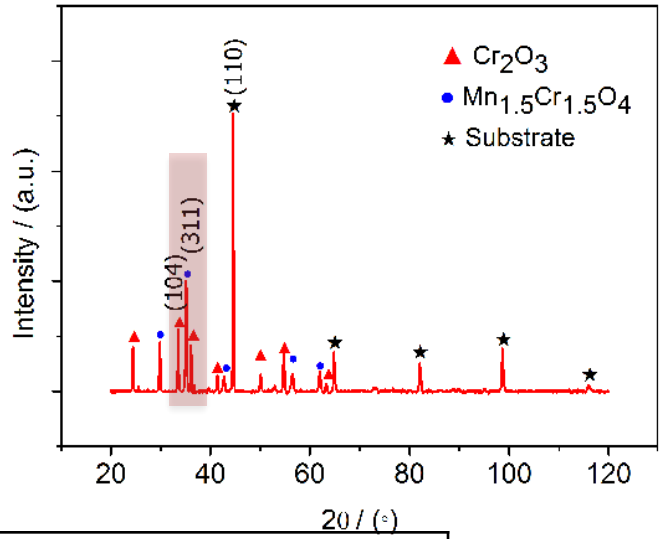




cross-section

XRD (AISI430)

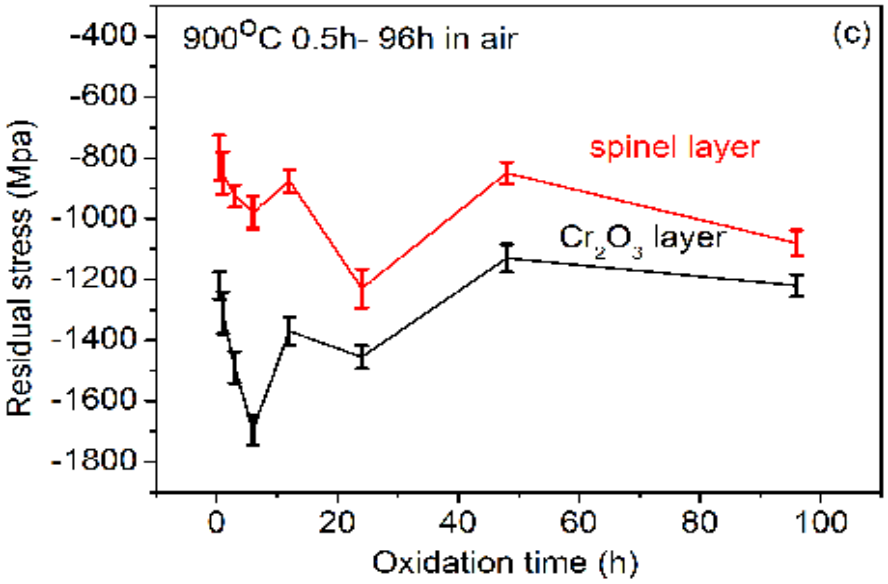


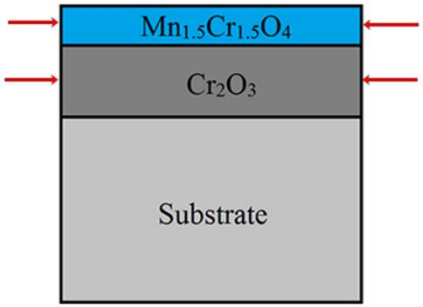


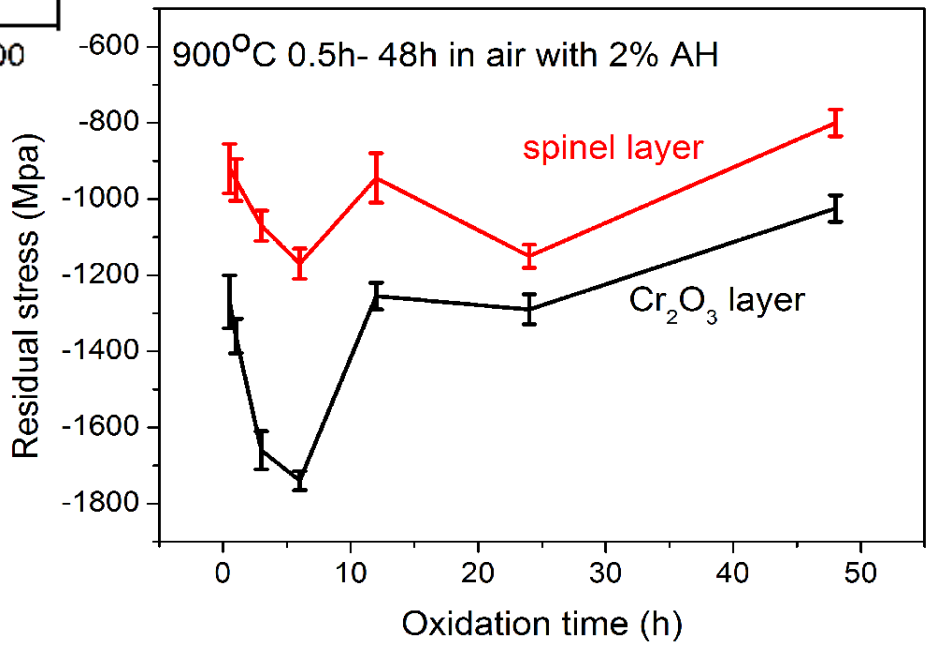
Analysis zone	2mm x 2mm
ψ angle amplitude	[-60° ; 60°]
Number of ψ angle	13
diffraction angle 2θ	33.6° for Cr_2O_3; 35.1° for $Mn_{1.5}Cr_{1.5}O_4$
2θ angle interval	[32.2-35] [33.7-36.5]
2θ step	0.04
Acquisition time	15 s /step
Precision	30 MPa
Young's modulas	Cr_2O_3 :280 GPa; $Mn_{1.5}Cr_{1.5}O_4$:250GPa
Poisson's coefficient	Cr_2O_3 :0.29 ; $Mn_{1.5}Cr_{1.5}O_4$: 0.27

stress analysis

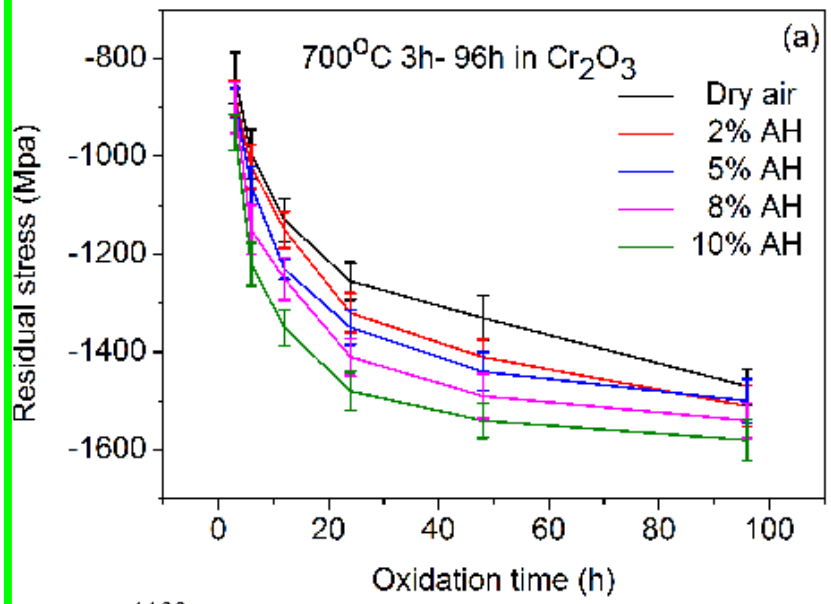
XRD (AISI430)

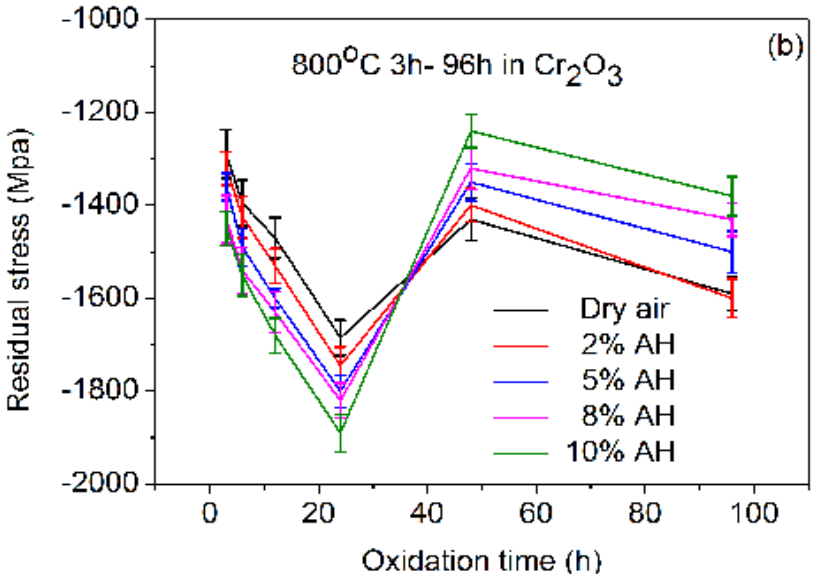

900°C 0.5h- 96h in air
(c)
spinel layer
Cr_2O_3 layer
Residual stress (Mpa)
Oxidation time (h)
-400
-600
-800
-1000
-1200
-1400
-1600
-1800
0
20
40
60
80
100

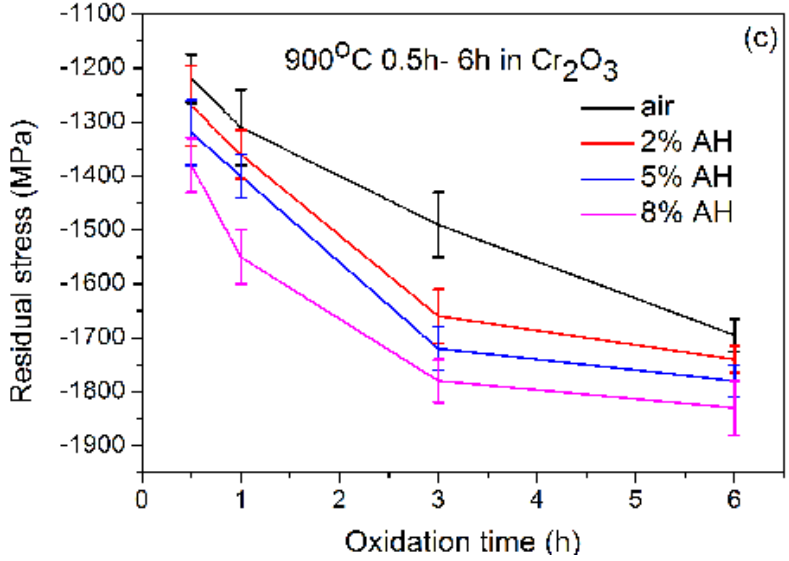

$Mn_{1.5}Cr_{1.5}O_4$
Cr_2O_3
Substrate

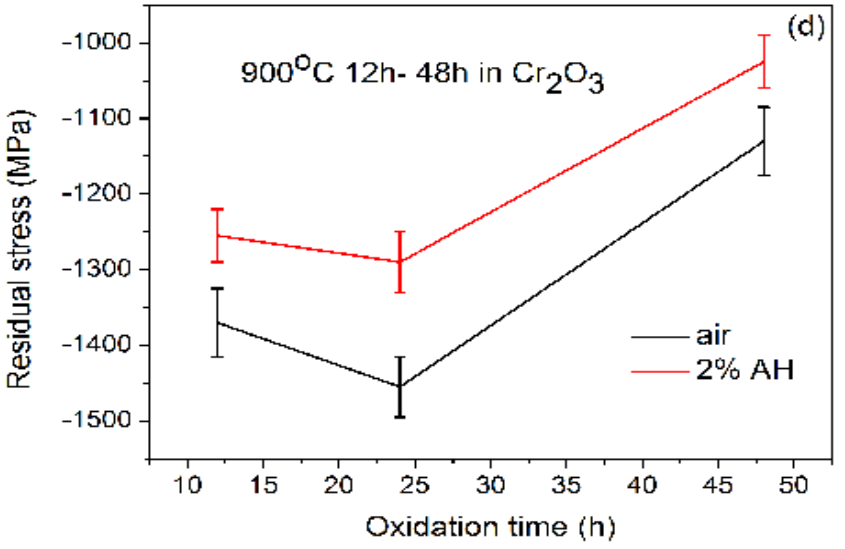

900°C 0.5h- 48h in air with 2% AH
spinel layer
Cr_2O_3 layer
Residual stress (Mpa)
Oxidation time (h)
-600
-800
-1000
-1200
-1400
-1600
-1800
0
10
20
30
40
50

XRD (AISI430)

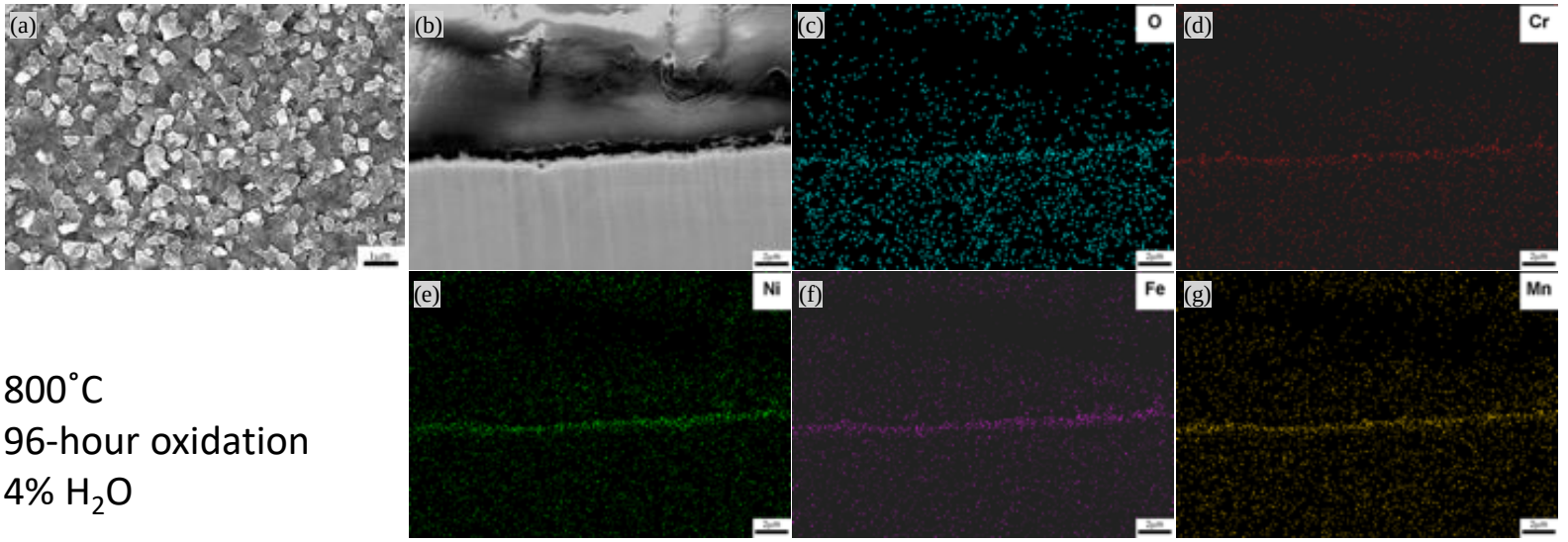

(a)
700°C 3h- 96h in Cr_2O_3
Dry air
2% AH
5% AH
8% AH
10% AH
Residual stress (Mpa)
Oxidation time (h)


(b)
800°C 3h- 96h in Cr_2O_3
Dry air
2% AH
5% AH
8% AH
10% AH
Residual stress (Mpa)
Oxidation time (h)

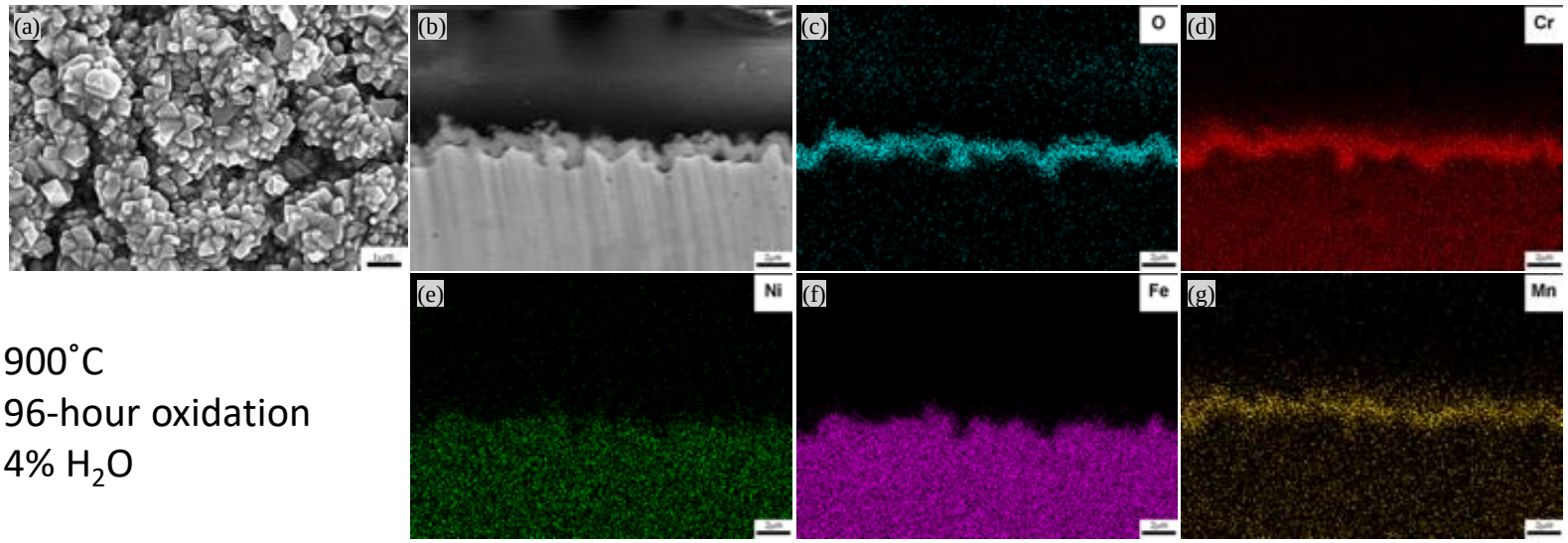

(c)
900°C 0.5h- 6h in Cr_2O_3
air
2% AH
5% AH
8% AH
Residual stress (MPa)
Oxidation time (h)


(d)
900°C 12h- 48h in Cr_2O_3
air
2% AH
Residual stress (MPa)
Oxidation time (h)

FEG-SEM (316L)

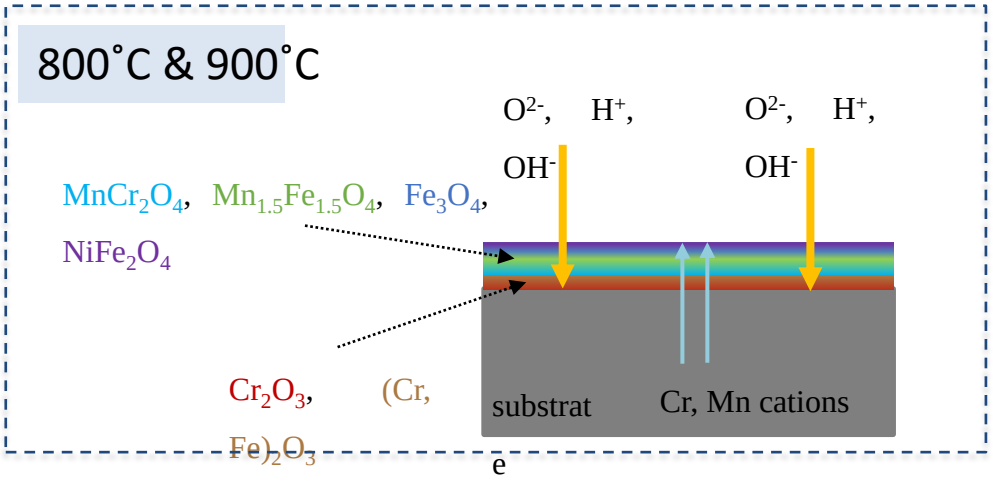


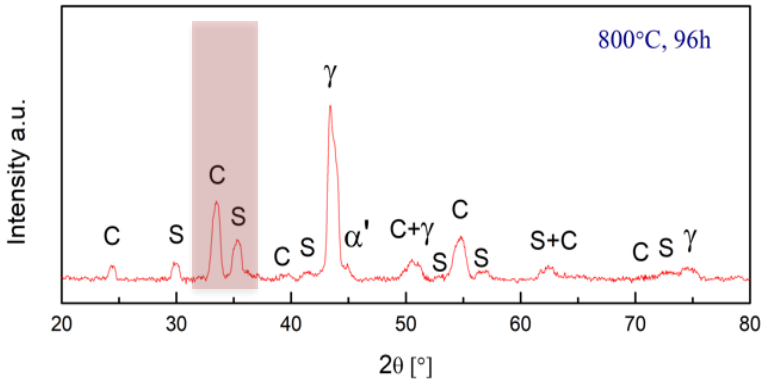
800˚C
96-hour oxidation
4% H_2O



900˚C
96-hour oxidation
4% H_2O

XRD (316L)

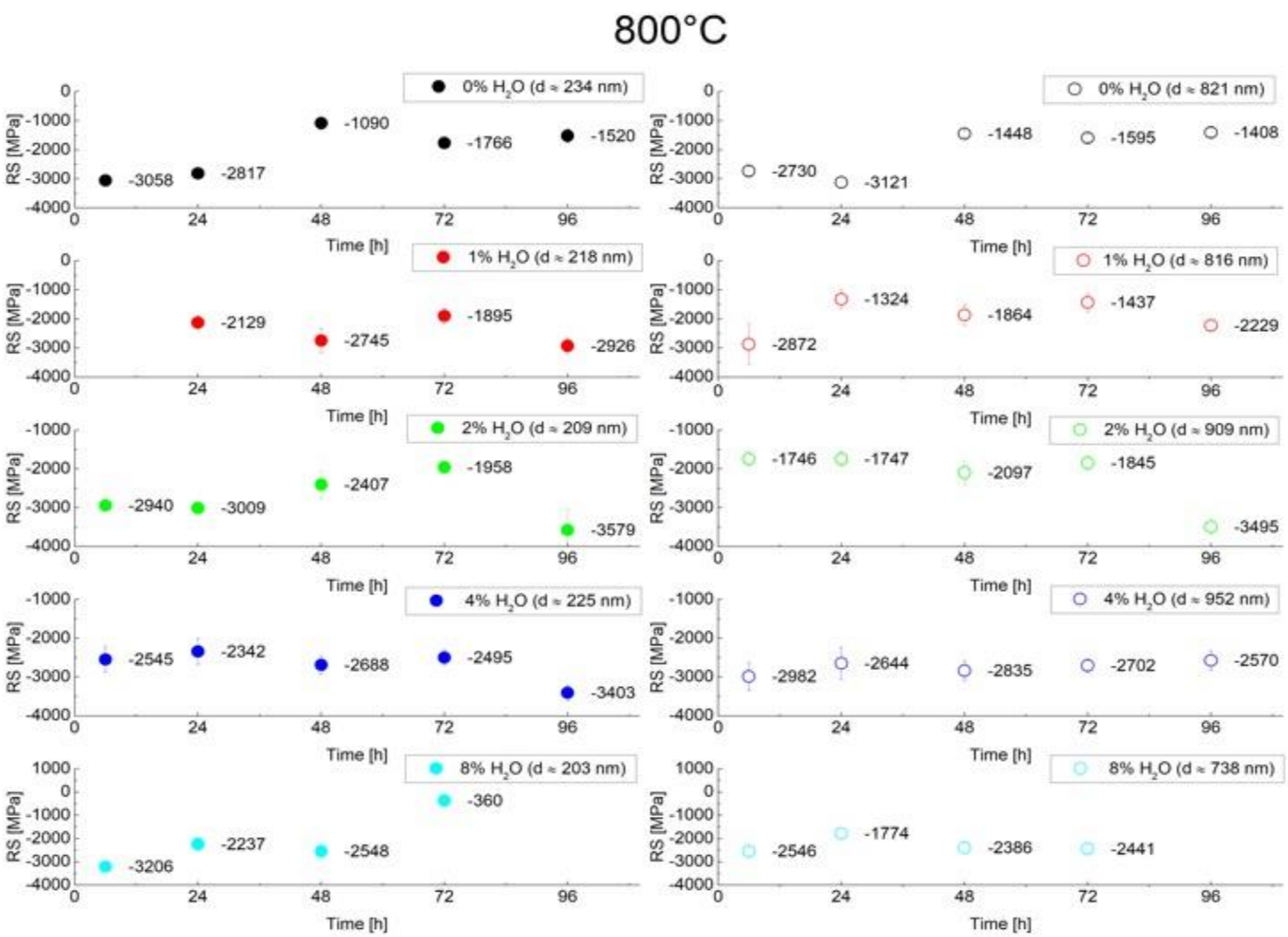




Analysis zone	2mm x 2mm
ψ angle amplitude	[-35˚ ; 35˚]
Number of ψ angle	13
diffraction angle 2θ	33.6˚ for Cr_2O_3 and $(Cr, Fe)_2O_3$; {1 0 4} 35.5˚ for $MnCr_2O_4$; {1 1 3}
2θ angle interval	[32.2-35] [33.7-36.5]
2θ step	0.04
Acquisition time	15 s /step
Precision	30 MPa
Young's modulas	Cr_2O_3 :280 GPa; $MnCr_2O_4$:177GPa
Poisson's coefficient	Cr_2O_3 :0.29 ; $MnCr_2O_4$: 0.32

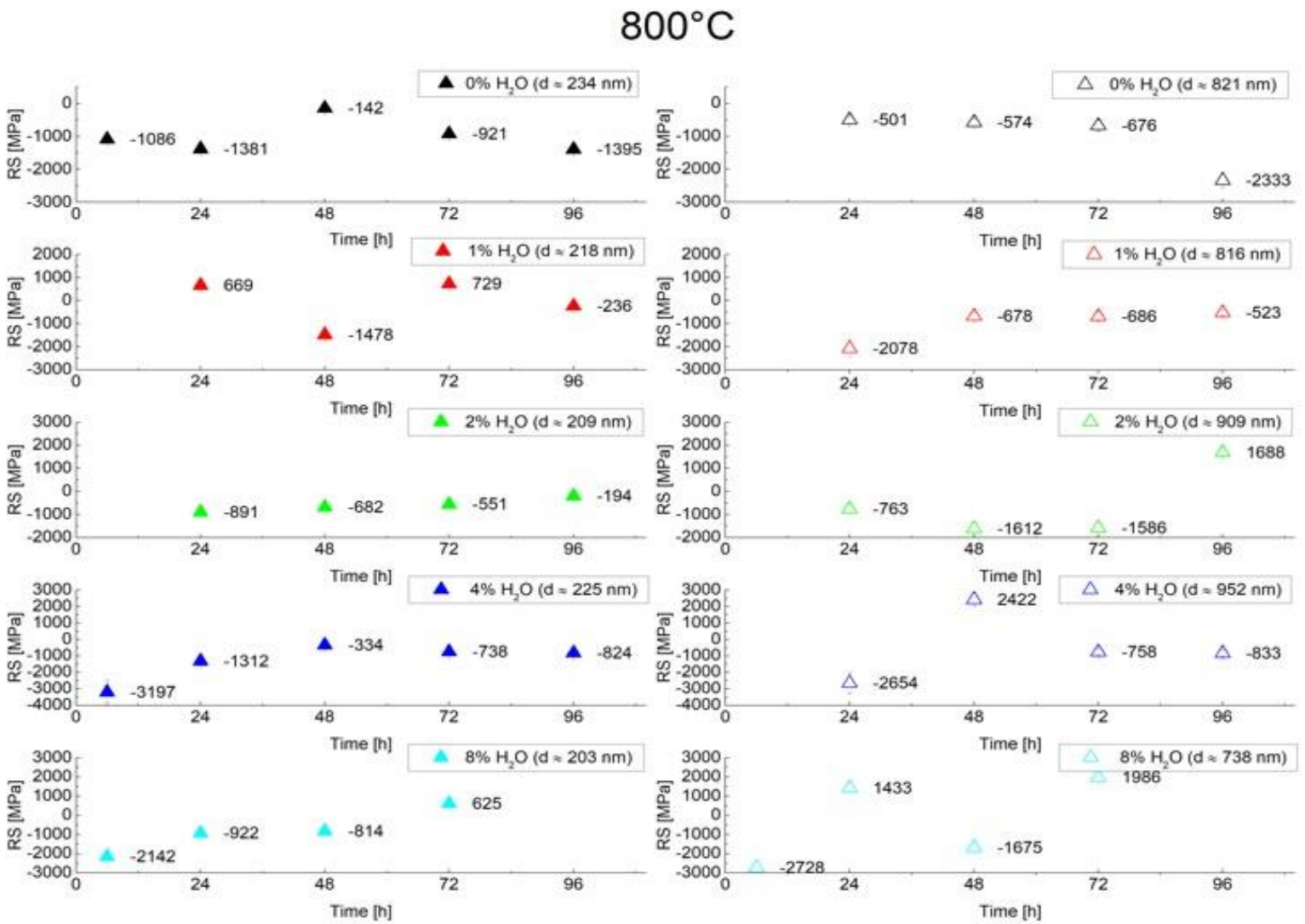
stress analysis

XRD (316L)



RS in Cr_2O_3 (internal layer)

XRD (316L)



RS in spinel (external layer)

Residual stress in oxide layers

$\sigma_{residual} = \sigma_{growth} + \sigma_{thermal} (+ \sigma_{chemical})$

σ_{growth} : weak compression
(by in-situ measurement)

$\sigma_{thermal}$: strong compression
(by calculation)

→→ $\sigma_{residual}$: strong compression

Conclusion

With humidity

- Oxidation kinetics are different for different temperature;
- XRD method leads to structure and residual stress information in two oxide layers;
- Residual stresses are in compression in two oxide layers.
- The residual stresses come from mainly thermal effect;
- The humidity and the oxidation temperature influences the residual stress levels.

Perspectives

- Residual stress analysis by in-situ XRD at high temperature.

- Growth stress analysis by in-situ deflexion at high temperature.

- Identification of –OH, -H or H_2 by IR spectroscopy or/and by XPS analysis

- Relationship between microstructure, oxidation kinetics and RS

Thanks for your attention !

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