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Mitigating Environmentally-Assisted Cracking Through Optimisation of Surface Condition – www.meactos.eu Horizon 2020

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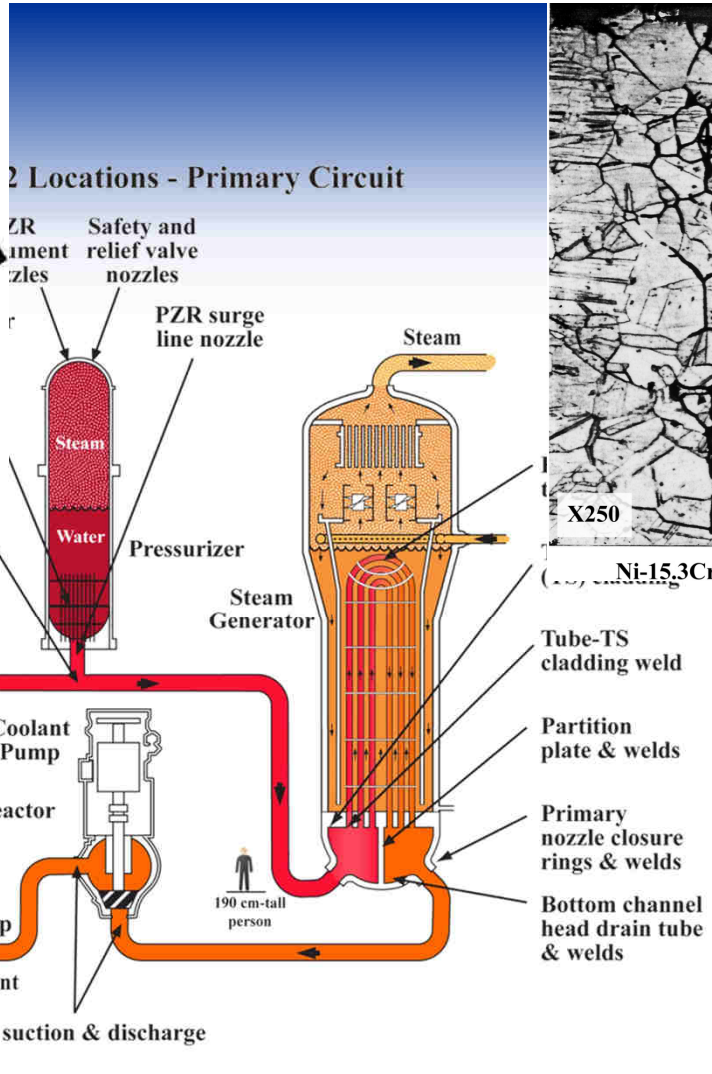

Ni-15.3Cr-7.0Fe-0.04C

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- **Stainless steels problemas encountered in in both BWRs and PWRs**
  - BWR: Sensitised 304 with high carbon content, 70s
  - BWR: Ti-stabilised, early 90s
  - Oxidising: EAC in RBMKs using Ti-stabilised
  - BWR: Non-sensitised 316L(N), later 90s
  - PWR: IGSCC in PWRs, increasing, now over 400 cases

OBJECTIVE

Study the applicability of advanced machining techniques, successfully applied in other industrial sectors such as aeronautics and automotive, to improve the resistance to crack initiation by EAC of austenitic alloys in LWR

Mitigating Environmentally-Assisted Cracking Through Optimisation of Surface Condition

15 laboratories from different eruopean countries

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Materials Performance Centre

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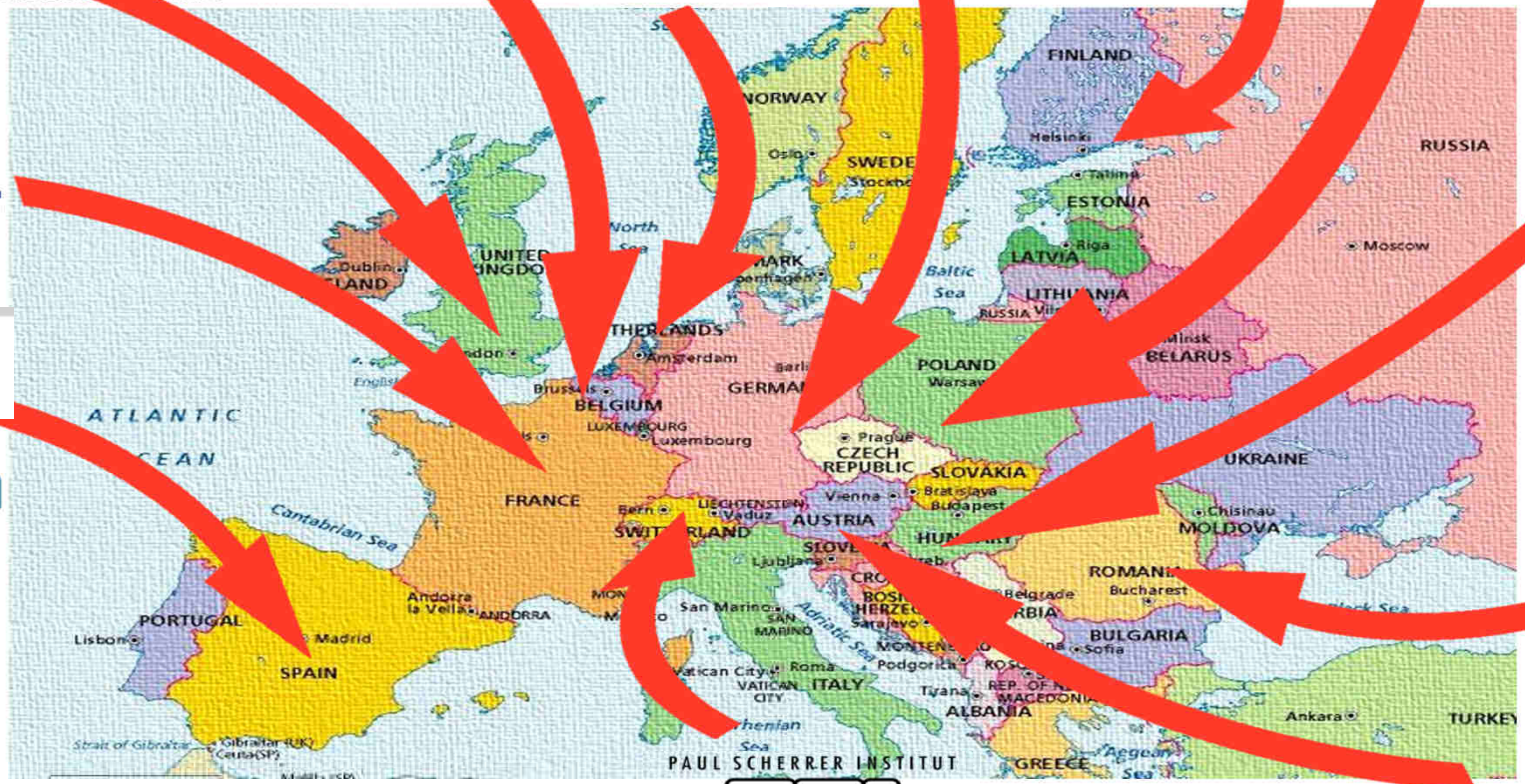
SLOVAK UNIVERSITY OF TECHNOLOGY IN BRATISLAVA



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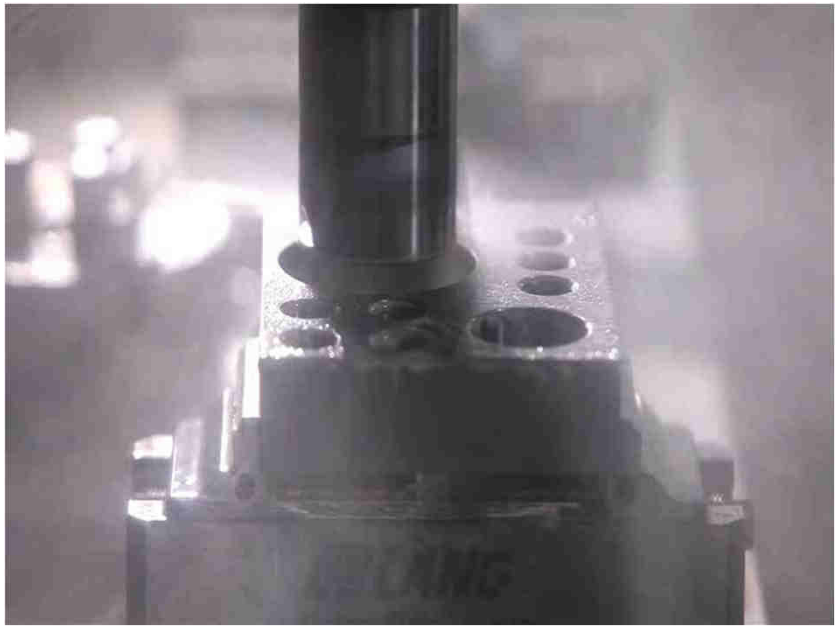
PSI

Advanced machining by chip removal: Advantages of using CO_2 as cutting fluid

- Previous studies have shown:
  - **Reduction of tensile residual stresses**
  - Elimination of cutting fluids
  - **Cleanliness (dry, clean part)**
  - **Improved surface integrity**
  - **Higher rate of heat removal**
  - Lower cutting zone temperatures
  - Improved tool life/increased productivity
  - **Improved surface finish**
  - Less impact on environment
  - Cost saving benefits
  - **Enabling/enhancing portable machining**
  - Controllable levels of cooling and lubrication to work zones that are difficult to manage and difficult to reach

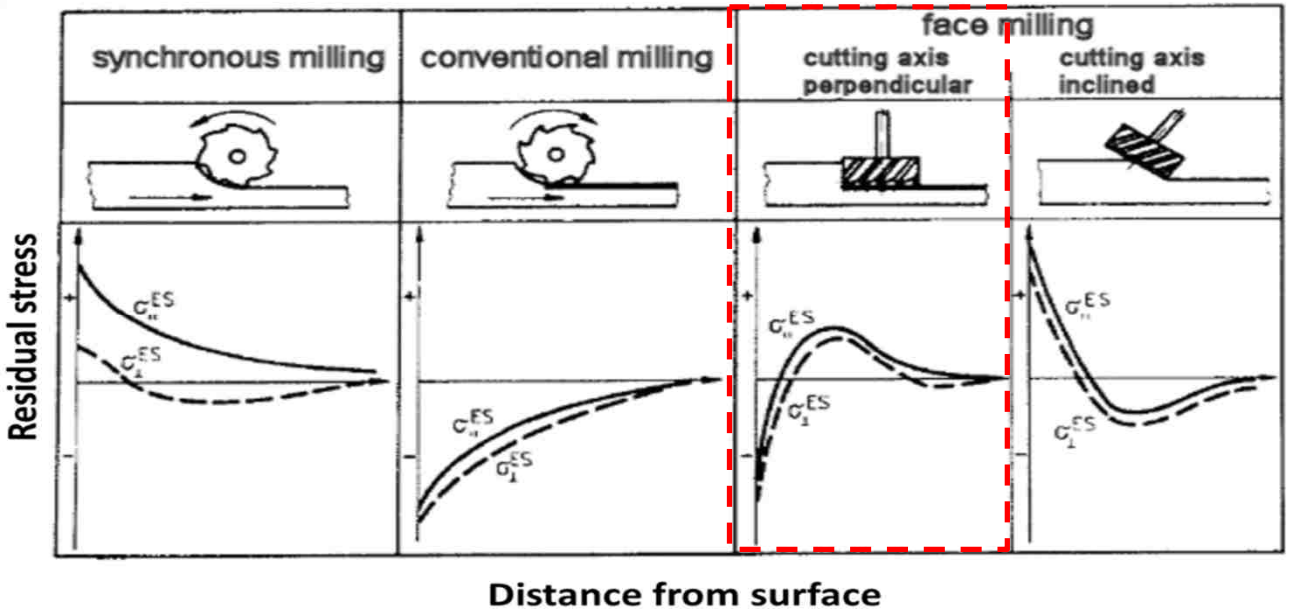
WP4: Surface machining & treatment - Machining

Classic



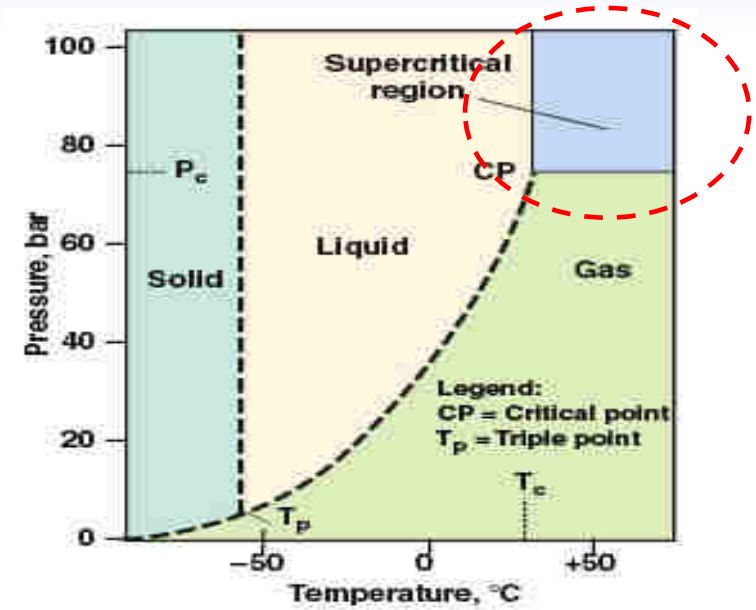
Advanced

Advenced Machining process: Face Milling with supercritical CO_2



- Most extensively machining procedure used in the industry
- Can produce a compresive layer close to the surface

Supercritical CO_2 + MQL



- Benefit from heat removal due to the expansion and absorption of heat from the supercritical fluid
- Possible benefit of the lubricating effect of liquid CO_2

Test Materials: Weld Metal (e.g. 182), Base Metal (e.g. A316L)

Experimentaton approach

WP4: Surface treatments
WP5 Characterization
WP6: EAC testing
WP7: Feeding the models

TASK 6.2

TASK 6.1

WP4 & WP6: Test matrix

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Material

- CW 316 L
- Alloy 182

Surface finish

- Industrial Conventional (STI)
- Advanced Machining (SAM)
- Surface Stress improvement (SP)

Surface modification

- Supercritical CO_2
- Supercritical CO_2 + MQL
- Shot peening
- Cavitaton (UHP)

Environments (BWR & PWR & SCW)

Screening phase
CERT test using tapered specimens

Material	Environment	Strain rate, 1/s
Alloy 182	BWR NWC, 288˚C	1×10^{-6}
		5×10^{-7}
		1×10^{-7}
	PWR, 320˚C, 1000 ppm B, 2 ppm Li	1×10^{-6}
		5×10^{-7}
		1×10^{-7}
Type A316L	BWR NWC, 288˚C	1×10^{-6}
		5×10^{-7}
		1×10^{-7}
	PWR, 320˚C, 1000 ppm B, 2 ppm Li	1×10^{-6}
		5×10^{-7}
		1×10^{-7}

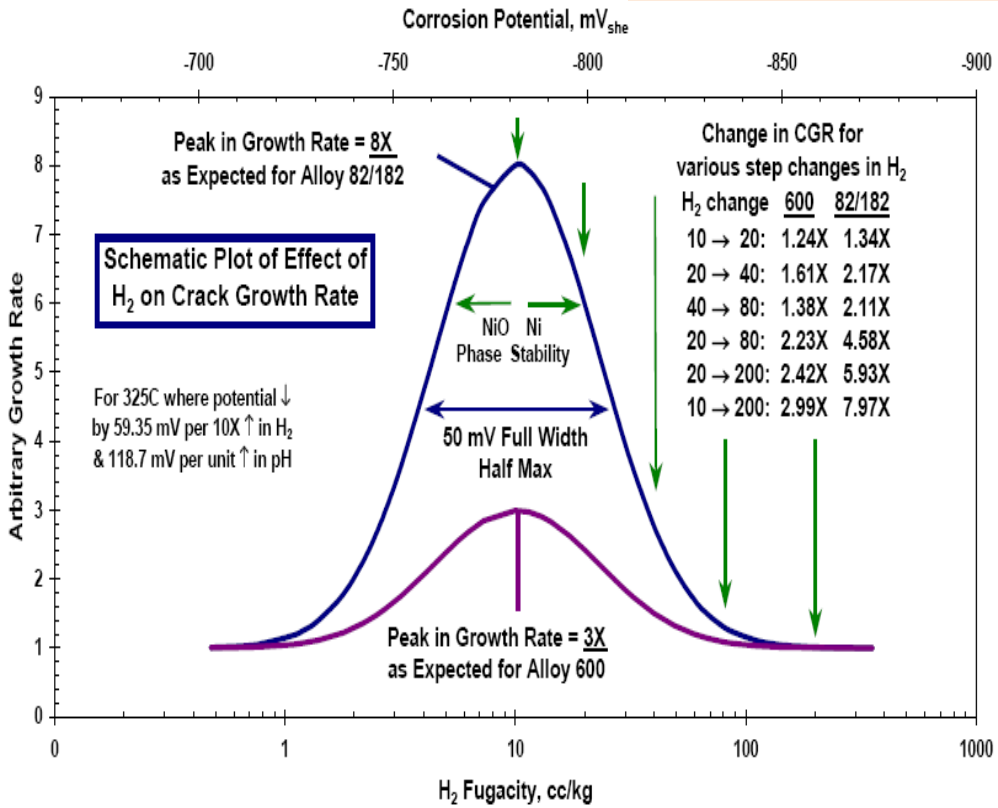
Verification & validation phase

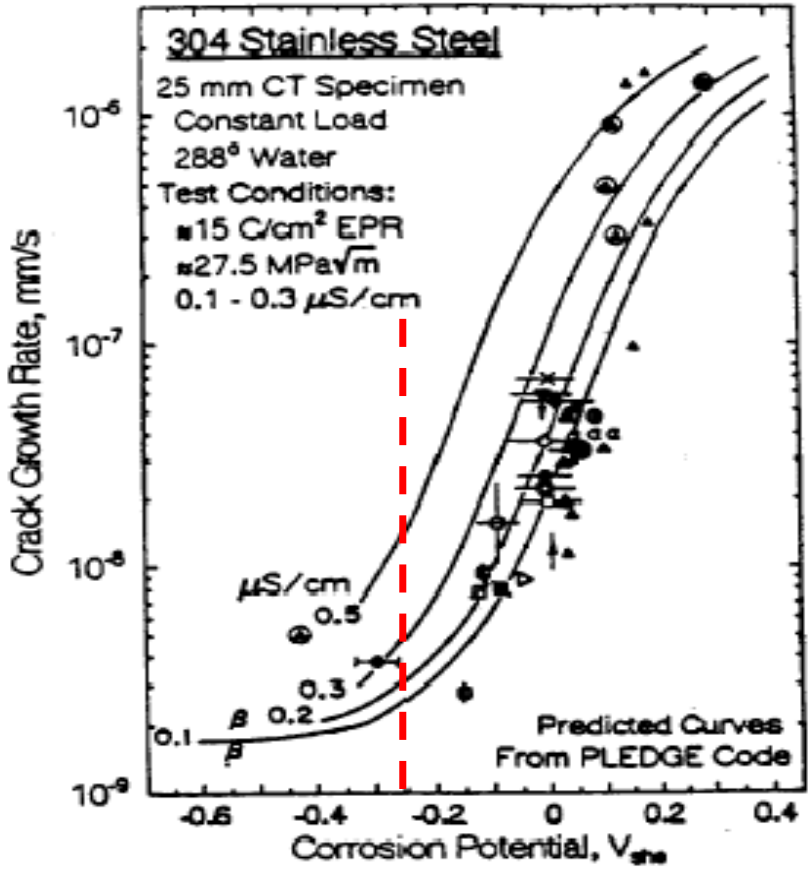
Constant load tests with smooth and notched specimens

A 182 & 316L in PWR environment

- Test temperature between 300-360°C
- [H_2] Dissolved in the Ni/NiO transition line

- Crack growth rate ↑↑
- Time to initiation ↓↓



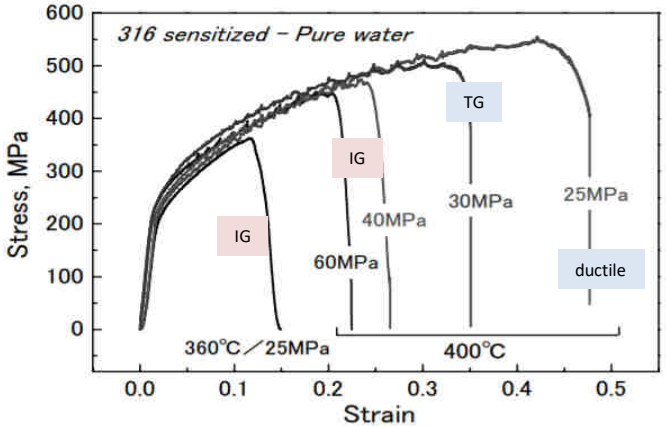


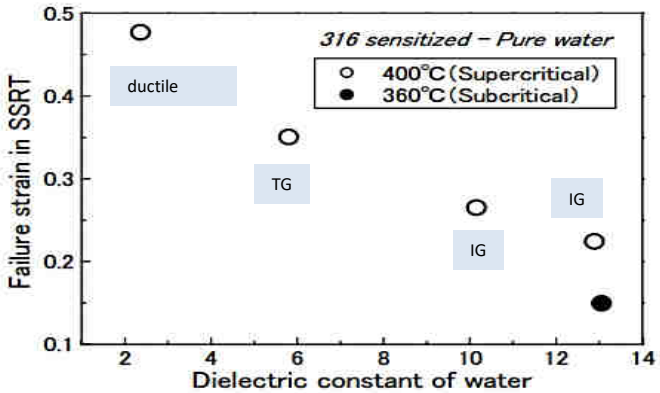
It is accepted that for Ecorr< -230mV crack growth rate decrease significantly

A hardness higher than 300-340 Hv, and cold work around 20% has been confirmed that promote cracking ➔ need to apply some degree of mechanical damage to the initial 316L to induce susceptibility

SCC of sensitized 316 SS in subcritical and SCW

Cracking susceptibility of sensitized 316 SS tested in pure water with 8 ppm of oxygen present a clear influence of the physical properties of water.



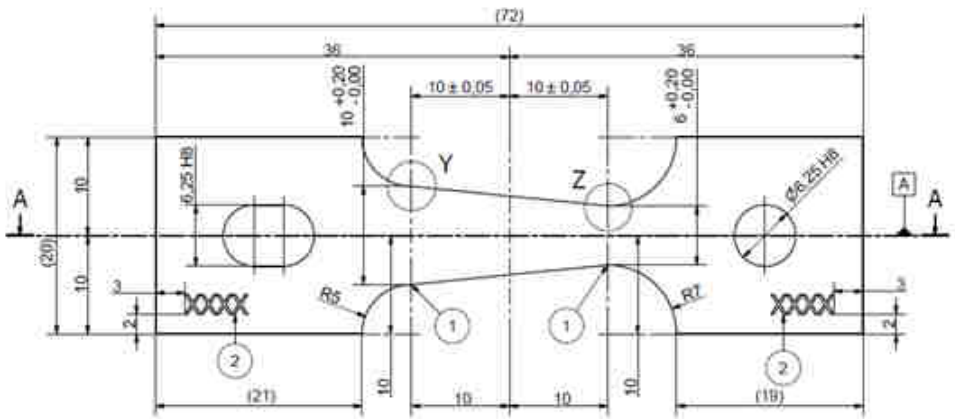


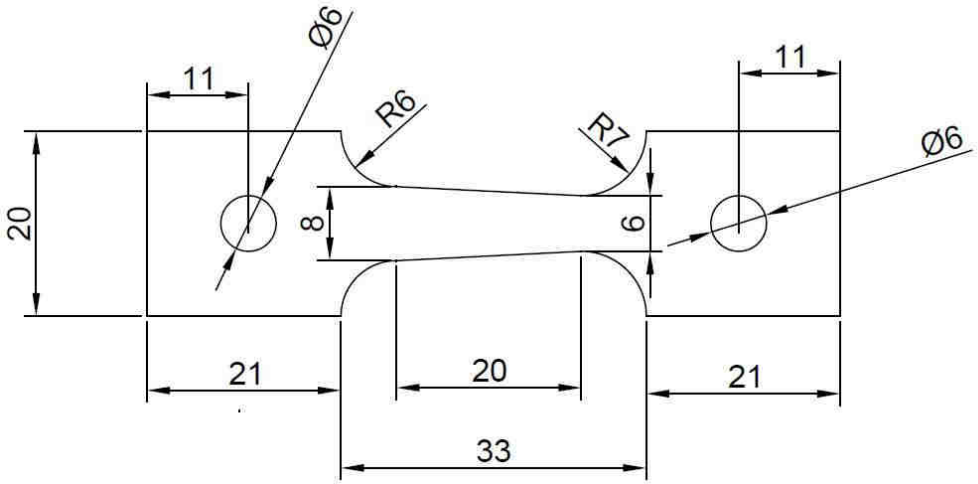
- *Cracking is enhanced under high dielectric constant conditions, in sub-critical and supercritical water.*
  - High dielectric constant : Ionization of metal is easier and solubility of metal oxides is higher ➔ Anodic dissolution of metal is higher
- The results give a strong evidence of the dissolution mechanism

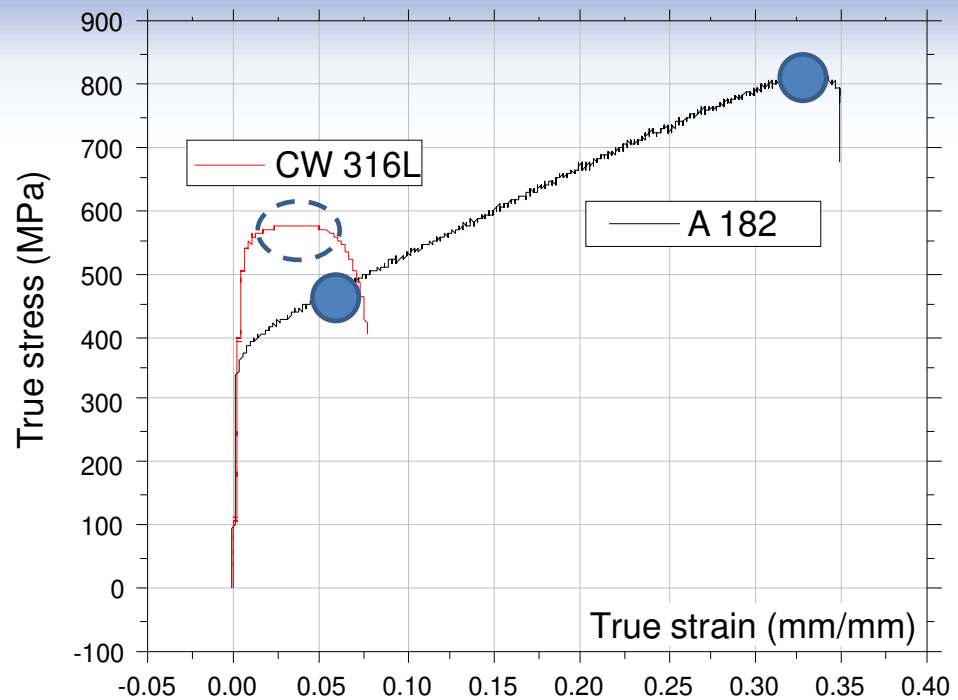
	T(ºC)	P(Mpa)	Density(g /mL-1)	Const. Diel.	Failure mode
SCW	400	25	0.16	2.4	ductile
SCW	400	30	0.36	5.9	TG
SCW	400	40	0.52	10.5	IG
SCW	400	60	0.61	13.3	IG
Water	360	25	0.59	13.4	IG

Watanabe, 2003

Tapered specimens: geometry adapted to characteristics of material tested





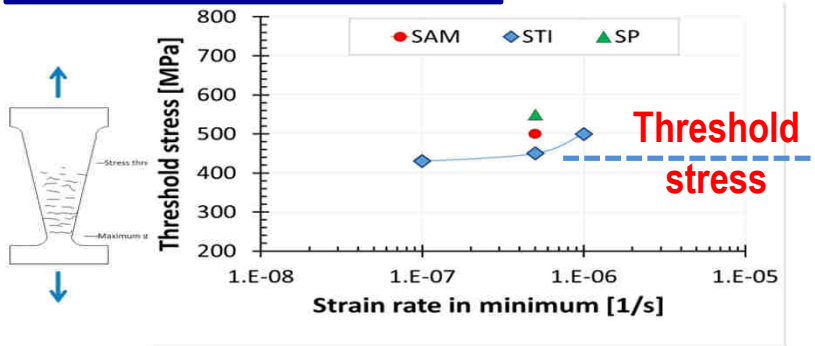


- Geometry of tapered length adjusted to get Ys to UTS in the tapered length
- Accepted to apply two different finishes to the specimens to test both in the same material condition/environment
  - 1 **Machined/treated side**
  - **1 Manually Polished (reference side) (RS)**

WP 6: Testing for EAC resistance under LWR cond.

- **Task 6.1 Screening**

CERTs in PWR/BWR



A 182 in PWR

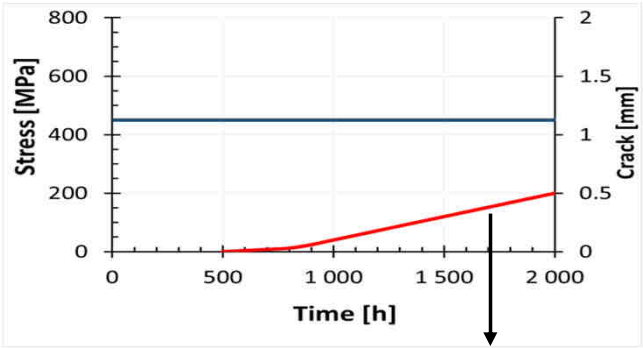
A 182 in BWR

316L in PWR

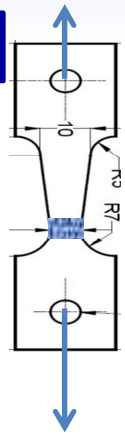
316L in BWR

- **Task 6.2 Verification & Validation**

Constant Load tests in accelerated conditions



Tapered



Threshold stress verification

Initiation time t_{ini}^{Acc}

PWR line:
PWR @ 320, 350-360°C
HSV @ 360, 400, 480°C

BWR line:
BWR @ 290, 360°C,
SCW @ 400, 480°C

182

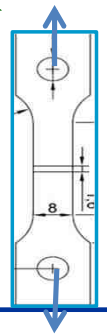
316L

$$t_{ini}^{Opr} = \eta \times t_{ini}^{Acc}$$

PWR

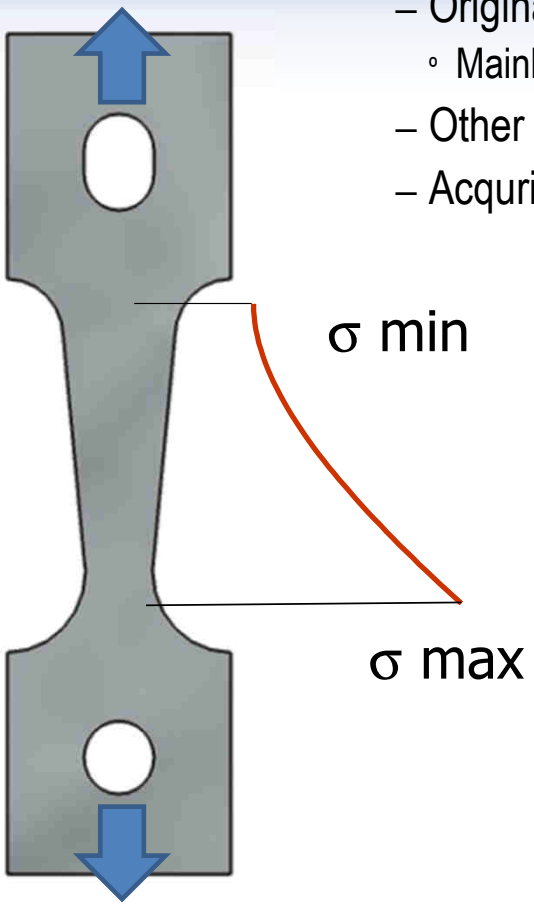
BWR

SENT



Tapered specimen: What is?

- Originally designed to obtain a stress gradient in the material in a single test
  - Mainly for Constant Load tests
- Other authors applied this geometry to SSRT, an CERT tests (dynamic)
- Acquried experience during previous MICRIN+ project using FLAT TAPERED specimens



Stress distribution

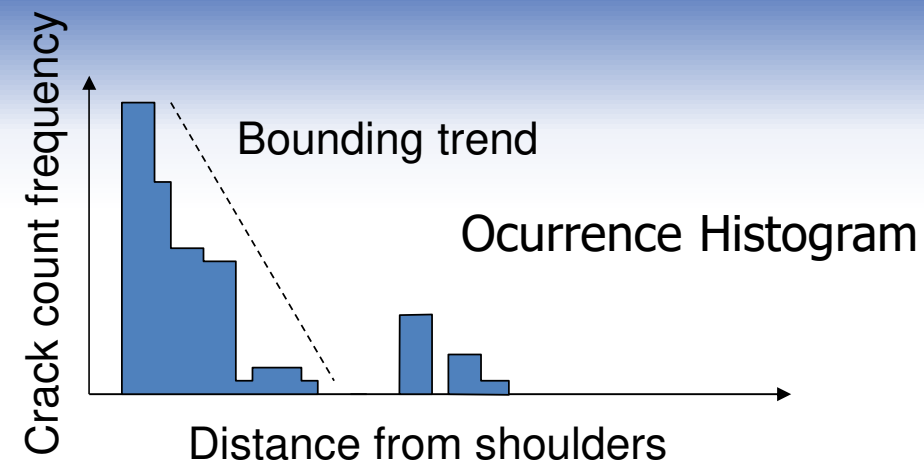
$$\frac{\sigma_{x=x}}{\sigma_{ys}} = \frac{\sigma_{x=0}}{\sigma_{ys}}\left[1 - \frac{x}{L}\left(1 - \frac{w_{x=0}}{w_{x=L}}\right)\right]$$

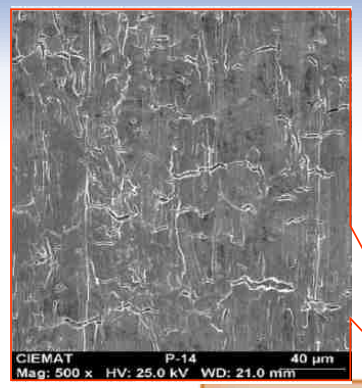
X=0 Minimum specimen width
L= tapered gage length
W= Width

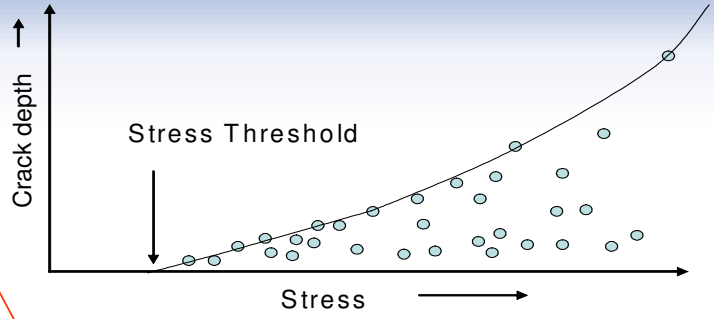
Stressed surfaces in contact with an aggressive environment will produce cracks, whose density and depth will depend on the stress level supported by the local section

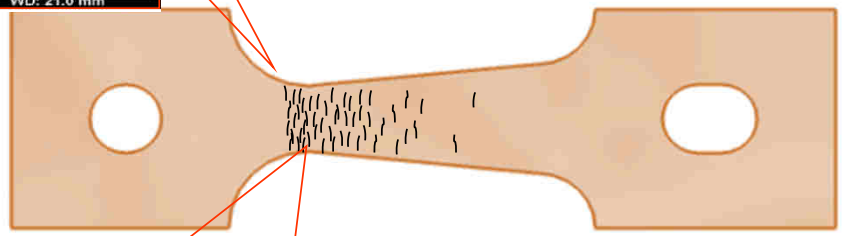
Designed by Jonas on 70's [O. Jonas. "Tapered Tensile Specimen for Stress Corrosion Threshold Stress Testing." Journal of Testing and Evaluation, Vol. 6, No. 1, 1978, pp. 40-47]

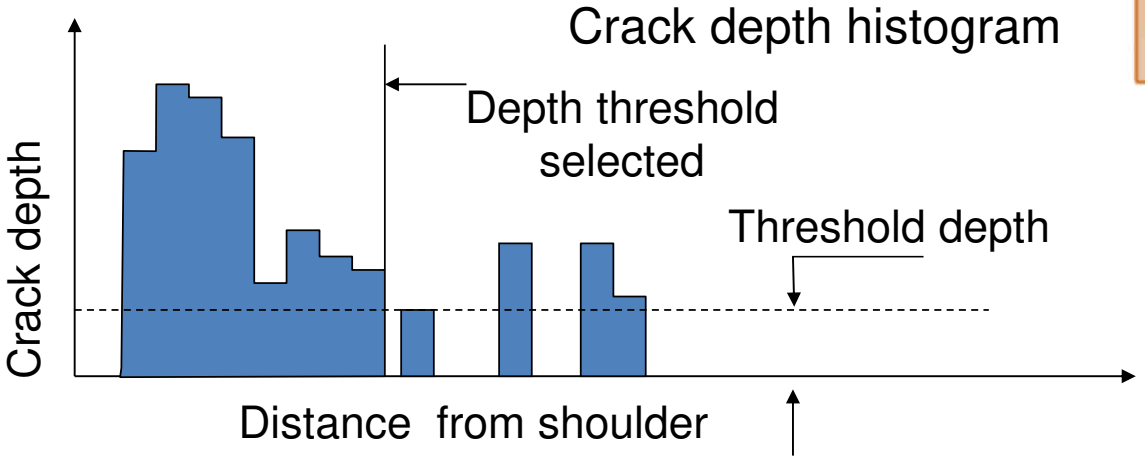
Tapered specimens: Crack distribution analysis

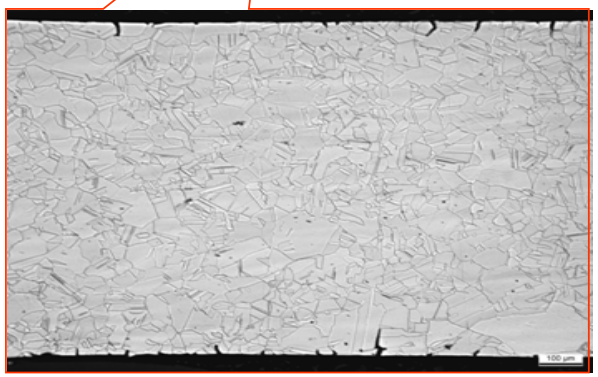










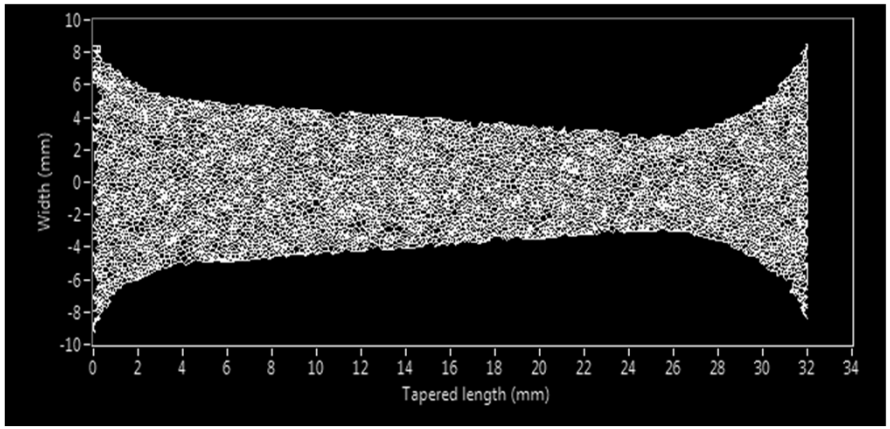


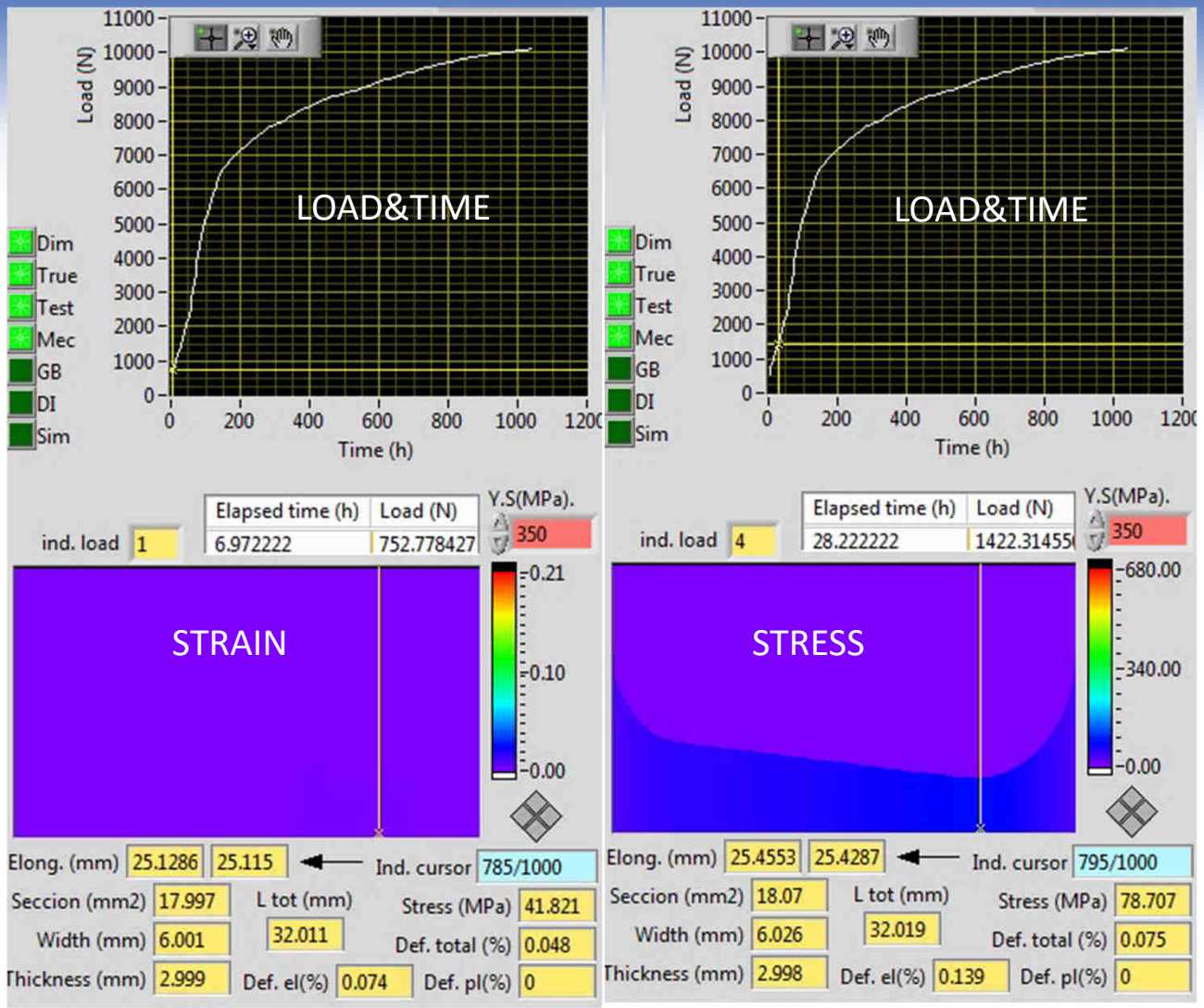
Cross section

Tapered specimens; Stress/strain and strain rate in a CERT/SSRT test

- When appling a constant displacement rate to the tapered section, a gradient of stresses/strains and strain rate is produced
- Hugh amount of information in one test
- Not obvious data interpretation ➔ models

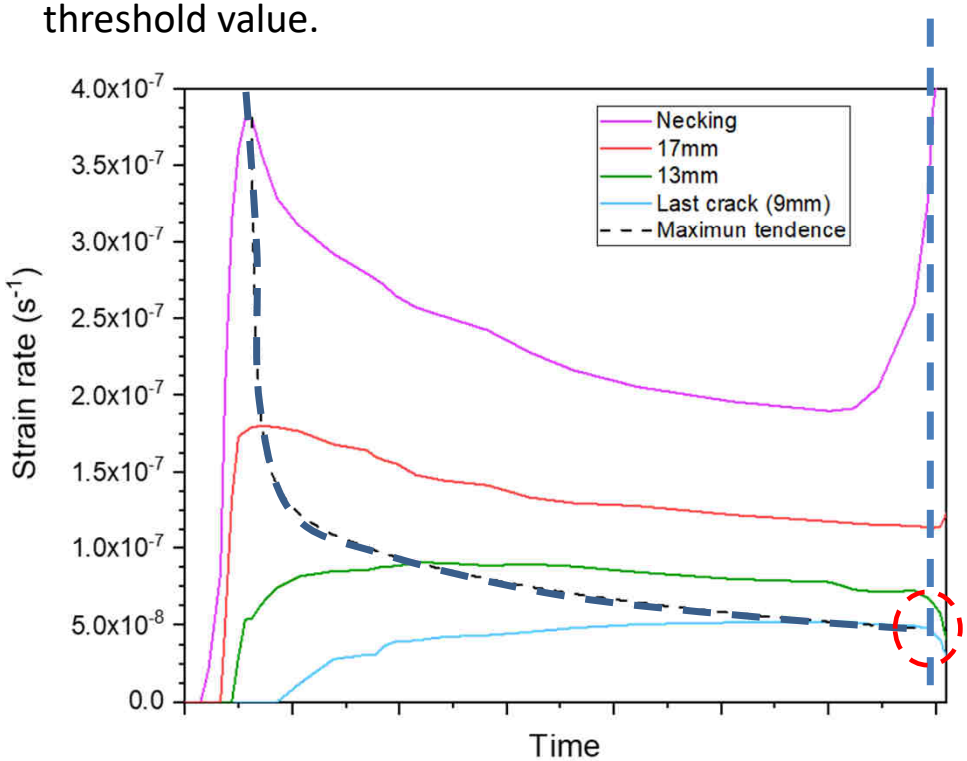
Simulation of strain of tapered length a test (Up to necking)



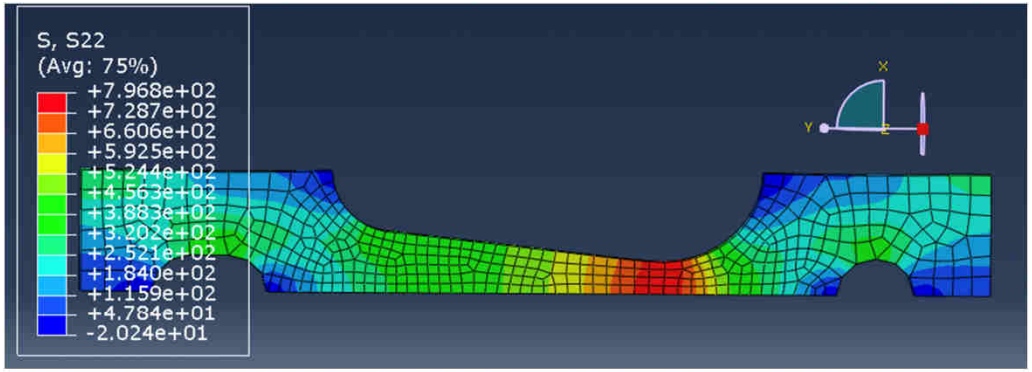


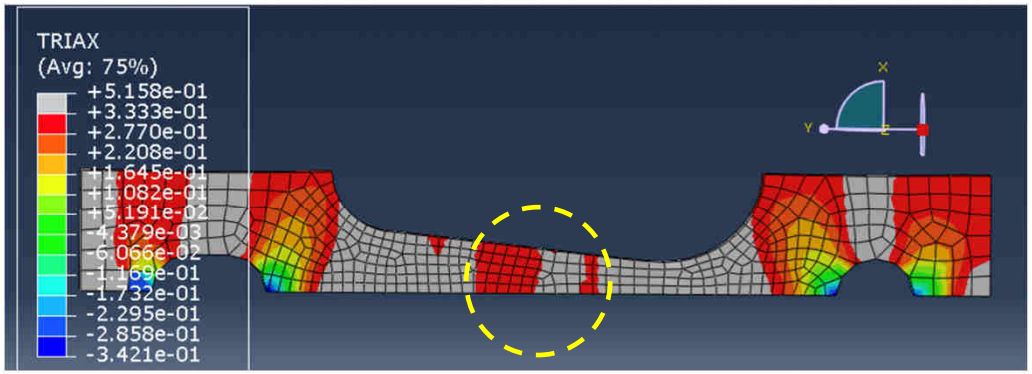
Tapered specimens: Numerical estimation of strain rate gradient along the length & triaxiality as factors for crack initiation

The intersection between the trend of the maximums and the vertical asymptote of the necking corresponds with the maximum of last crack strain rate-time curve, can be a criterion to determine a purely mechanic stress threshold value.



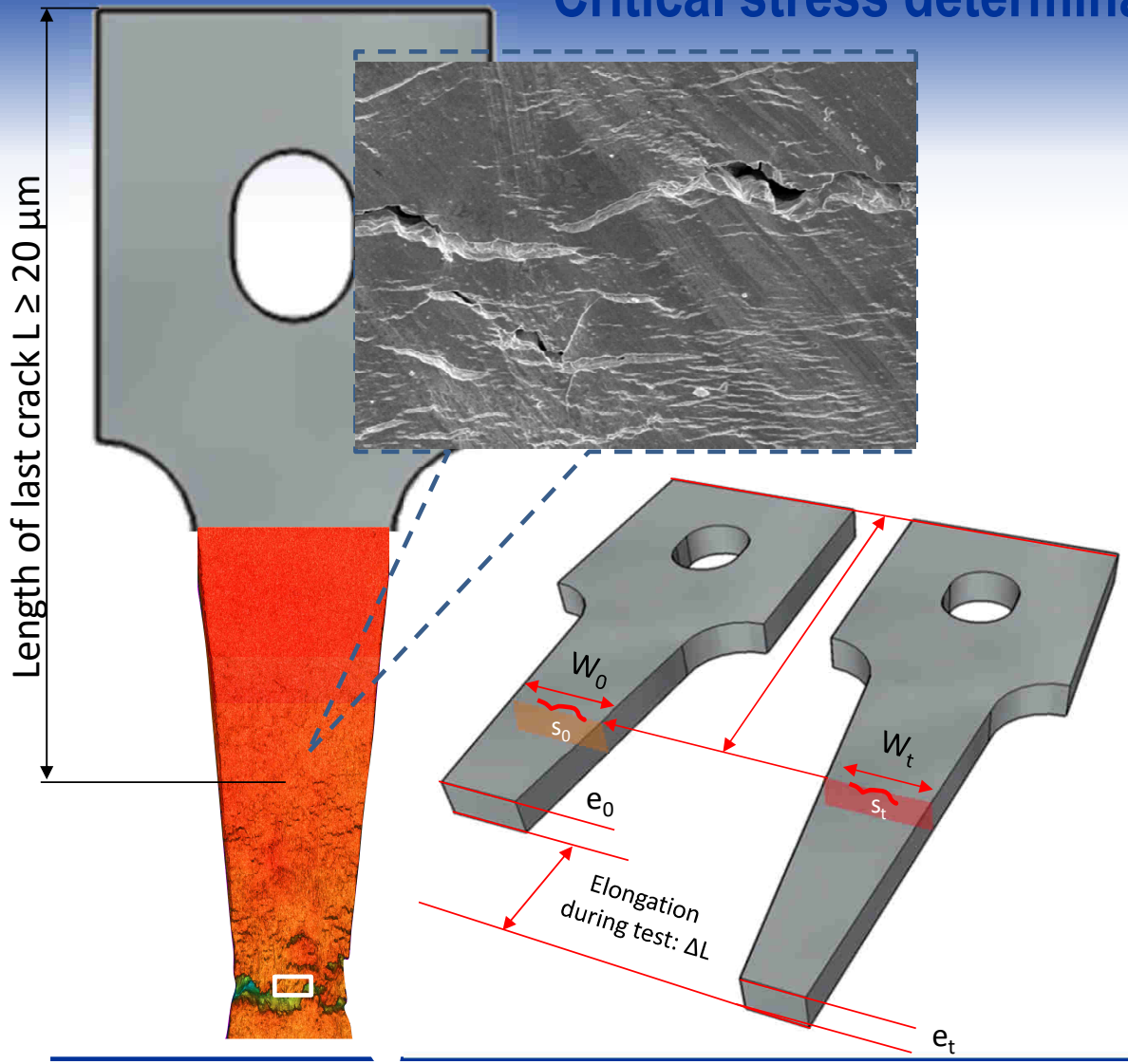
Equivalent stress distribution





Triaxiality as criterion for crack initiation

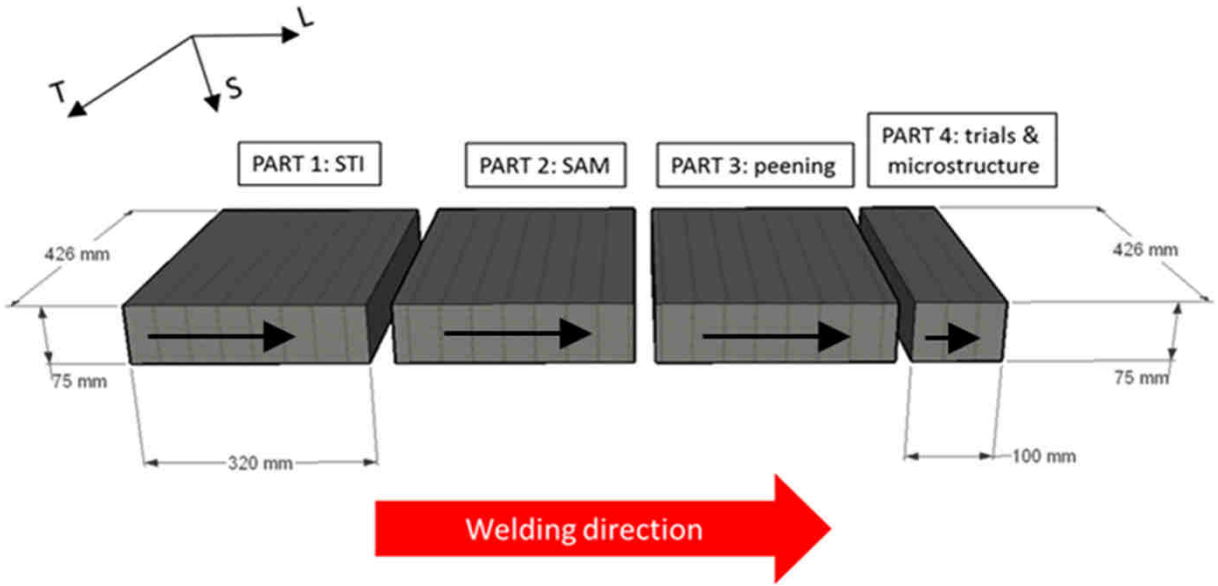
Critical stress determination



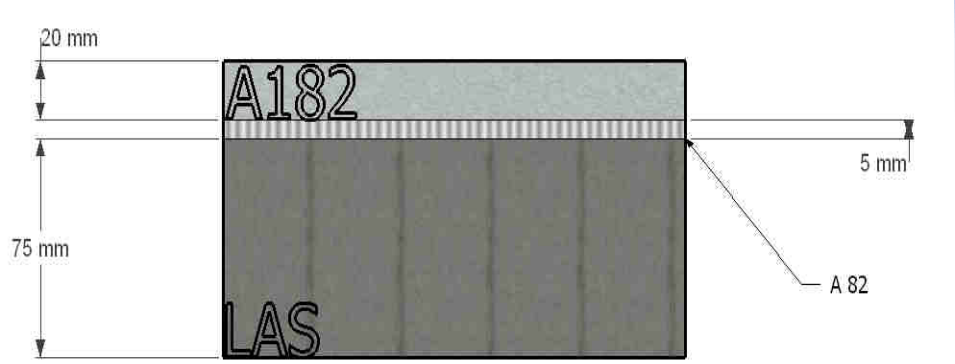
- Examination by SEM
  - Morphology associated to EAC
    - The oxidation layer was not chemically removed to avoid false cracks indications due to the effect of the reagent
    - Small pure mechanical cracks not easy identifiables
    - Not clearly opened cracks difficult to found
  - Location of the last crack L>20 µm
    - Original Specimen width at position of last crack
    - Thickness measurement at this position
  - Estimation of the resistant area
    - $W_0 \times e_t$
  - **Critical Stress = $F / (W_0 \times e_t)$**

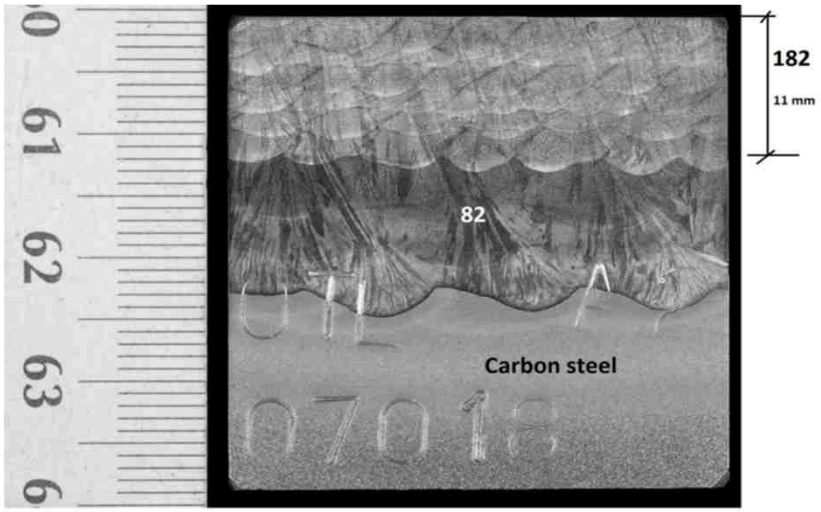
Coupons fabrication

A 182



Weld overlay of A82 and A182 was performed by ENSA



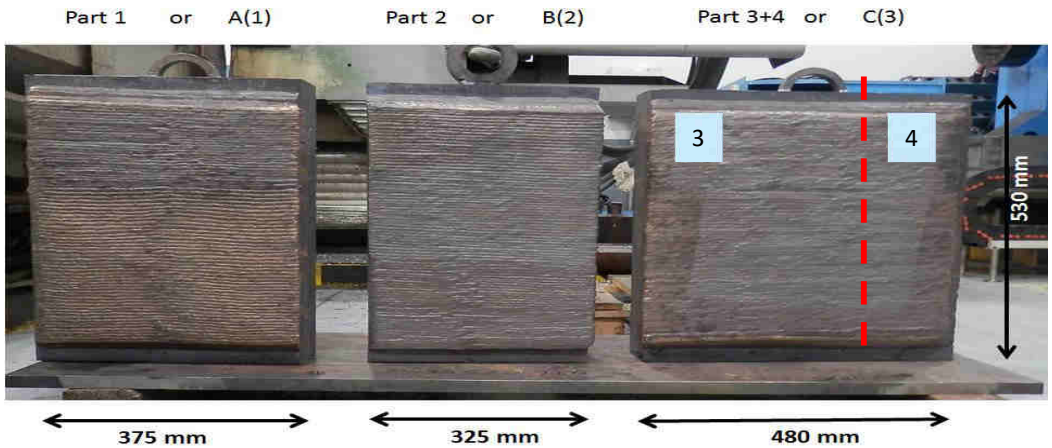


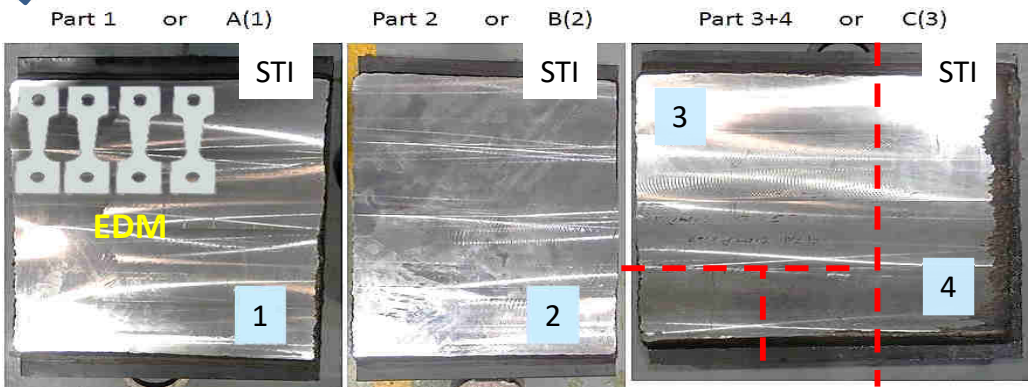
Metallographical cross section from Part 1.

Surface Machining treatment

A 182

Pieces were milled (STI) and squared at ENSA





Welding & Machining direction

STI

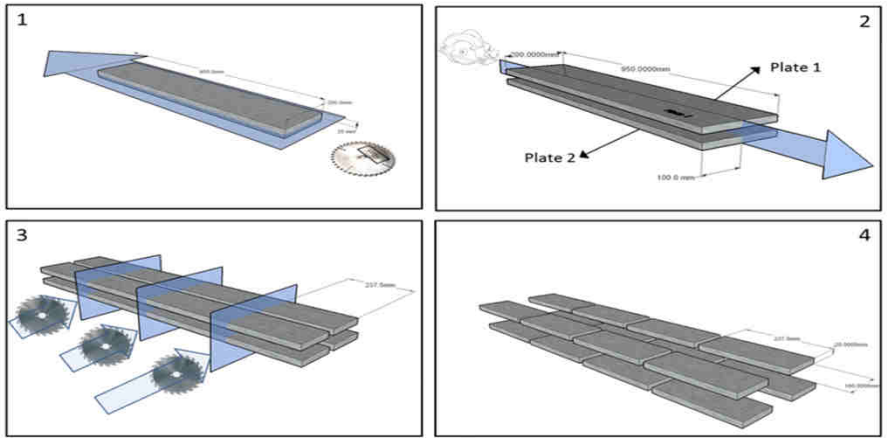
Sup CO_2

SP & CAV P

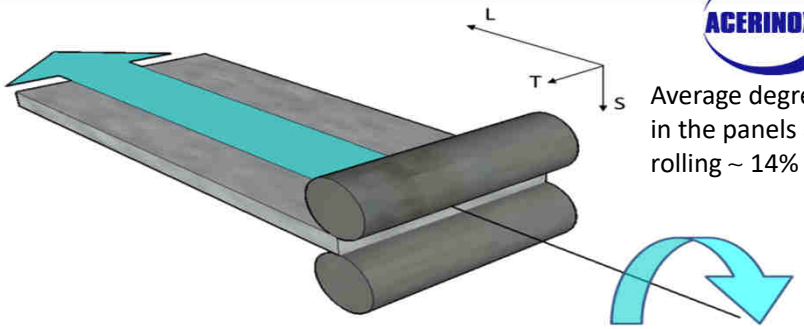
Opposite faces were polished (P2000)

Surface Machining treatment

316 L

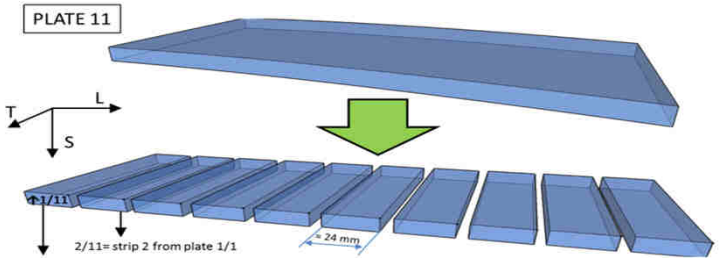


To facilitate subsequent cold work, 10mm thick panels were machined from the plate out of the upper and lower surfaces

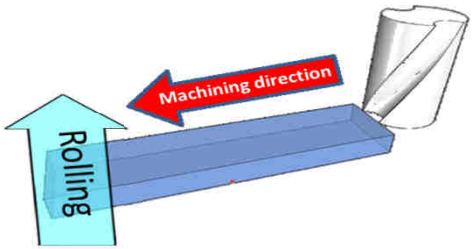


Average degree of strain in the panels after cold rolling ~ 14%

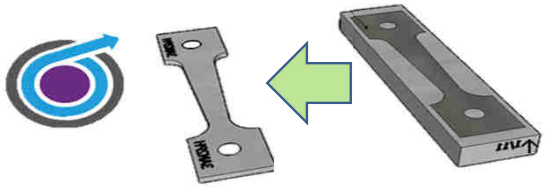
The panels were bent due to the rolling



Panels were cut by means of laser cutting into smaller pieces. Half of the pieces were milled in ENSA



EDM

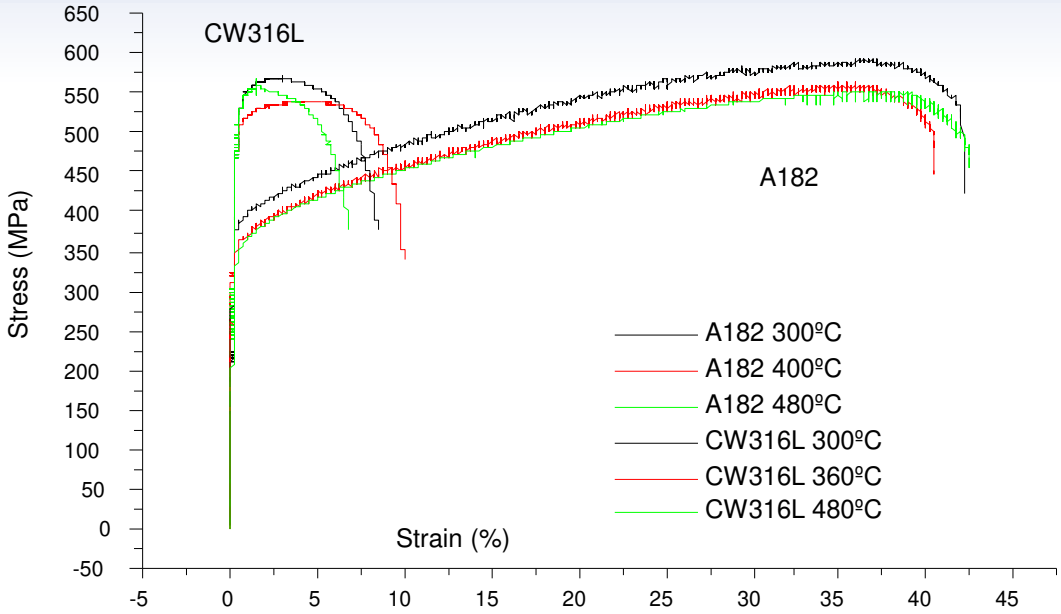


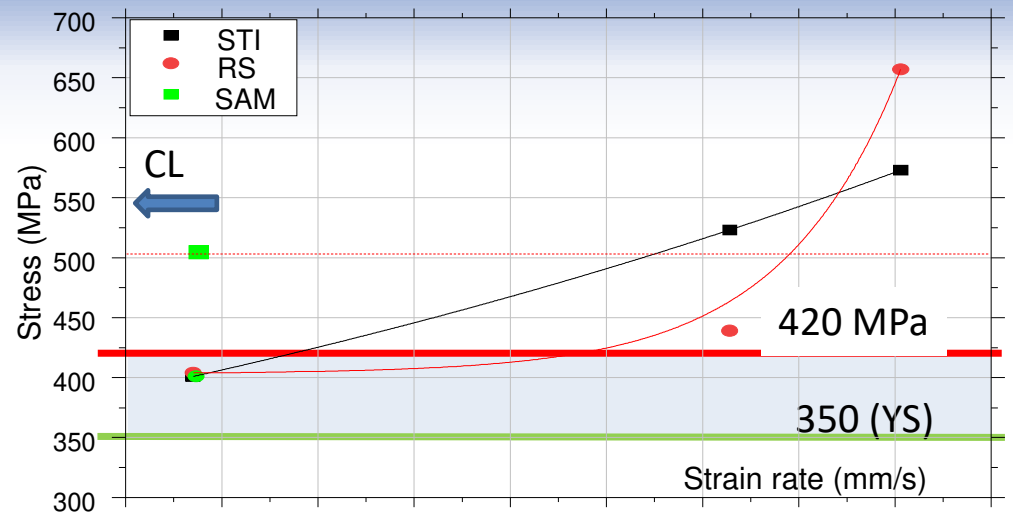
STI

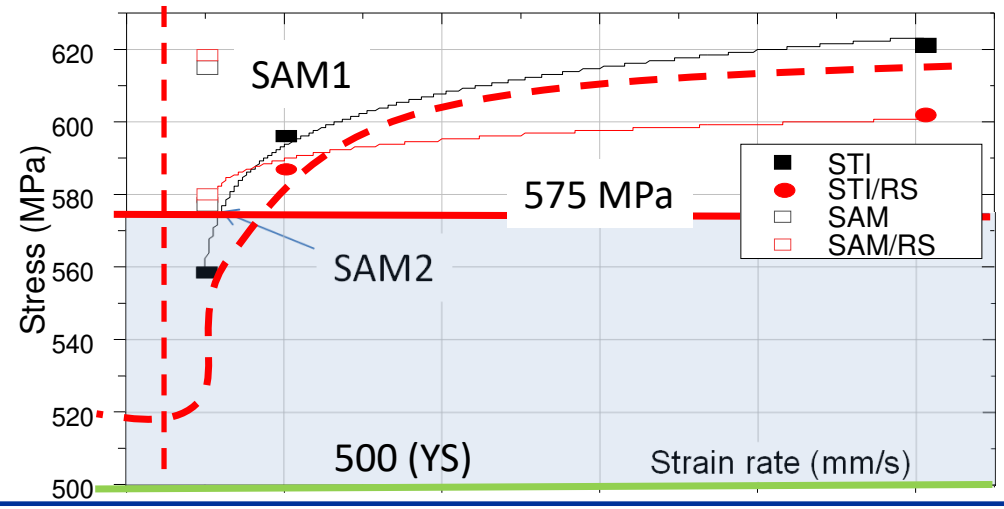
Sup CO_2

Sup CO_2+MQL

Results: A-182 & CW316L
(Stress threshold for EAC initiation)

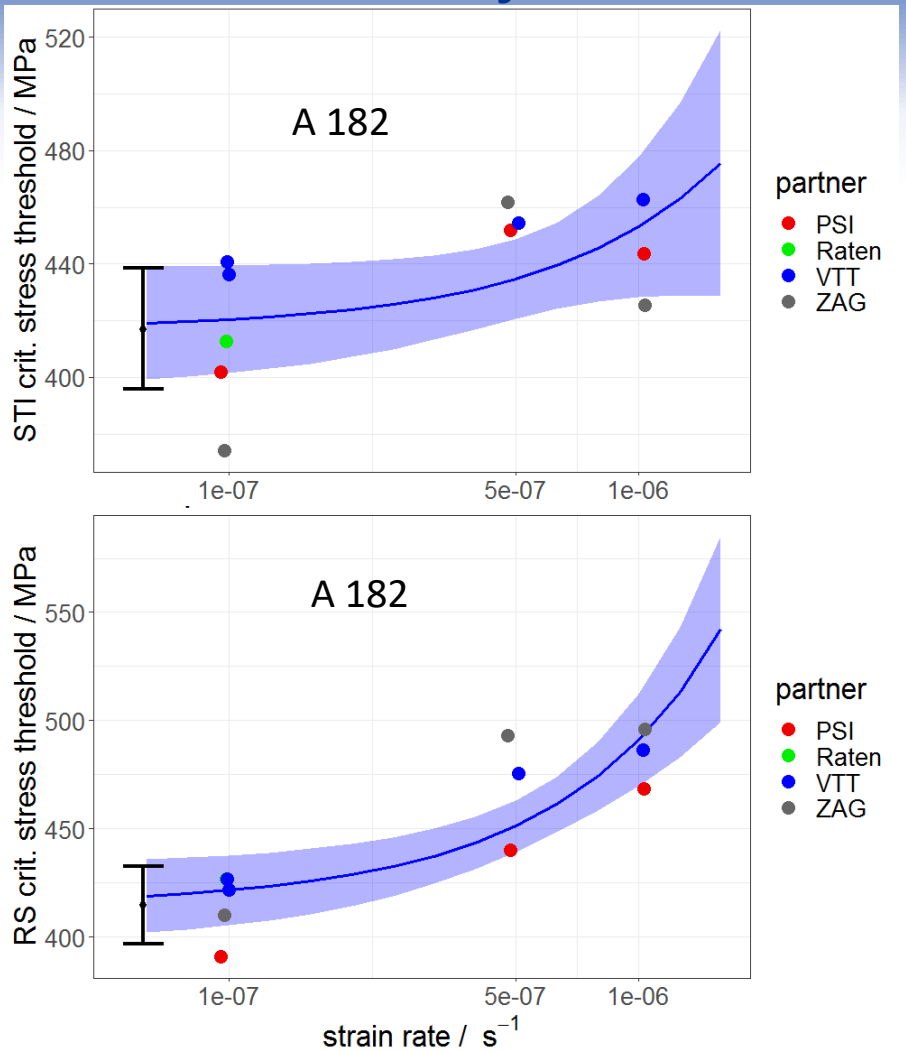


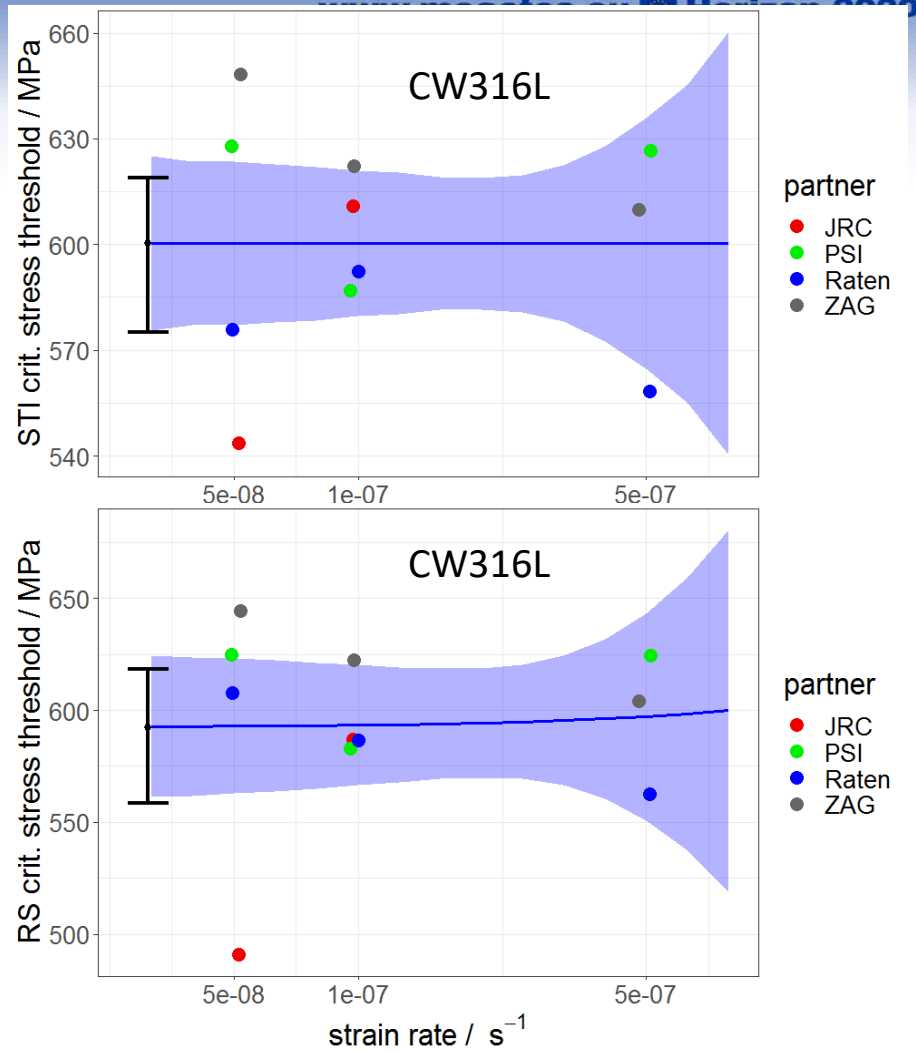




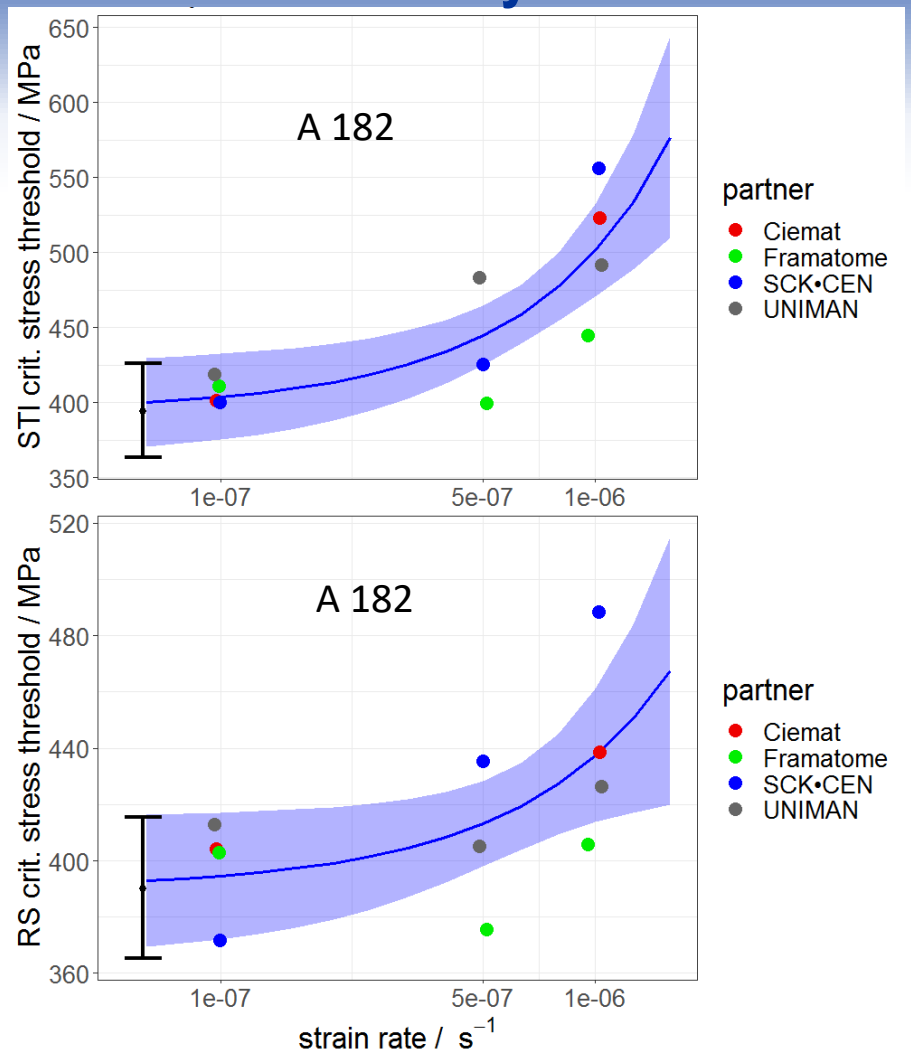
- Both materials show similar UTS although with evident difference in ductility. A 182 shows higher plasticity and hardenability compared to CW316L,
- ➔ The critical stress values as a function of strain rate for A 182 shows an asymptotic behavior for CL, CW316L it seems to have no asymptotes, At least for the applied cold work.
  - The profile of the curve towards the constant load limit is sigmoidal, so it would show a threshold at deformation rates lower than those used.
  - There is NO threshold value.
  - Cracking is possible at any load value.

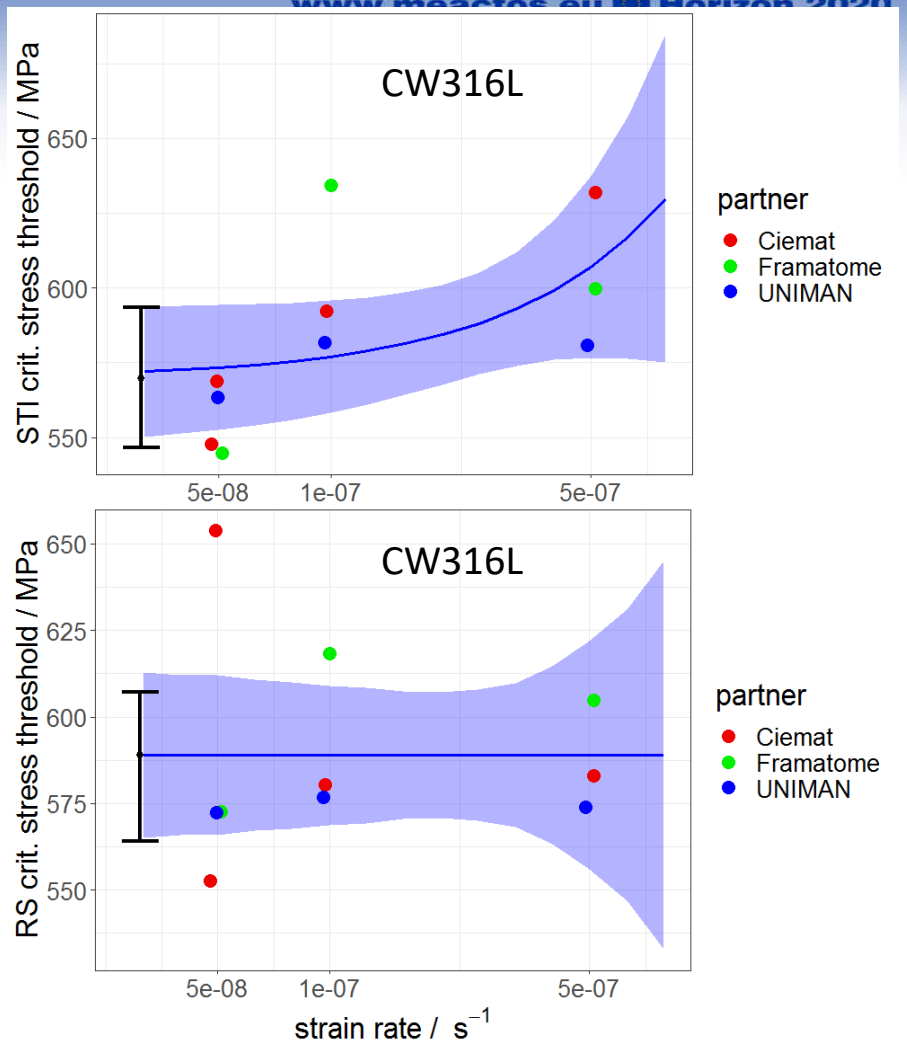
Results of threshold by environment: BWR





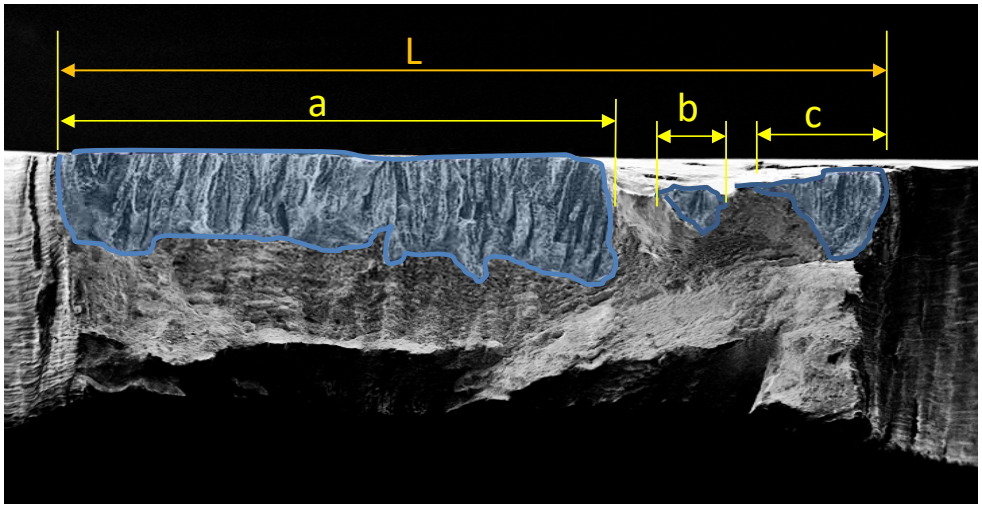
Results of threshold by environment: PWR





Engagement of interdendritic morphology in fracture surface of A 182

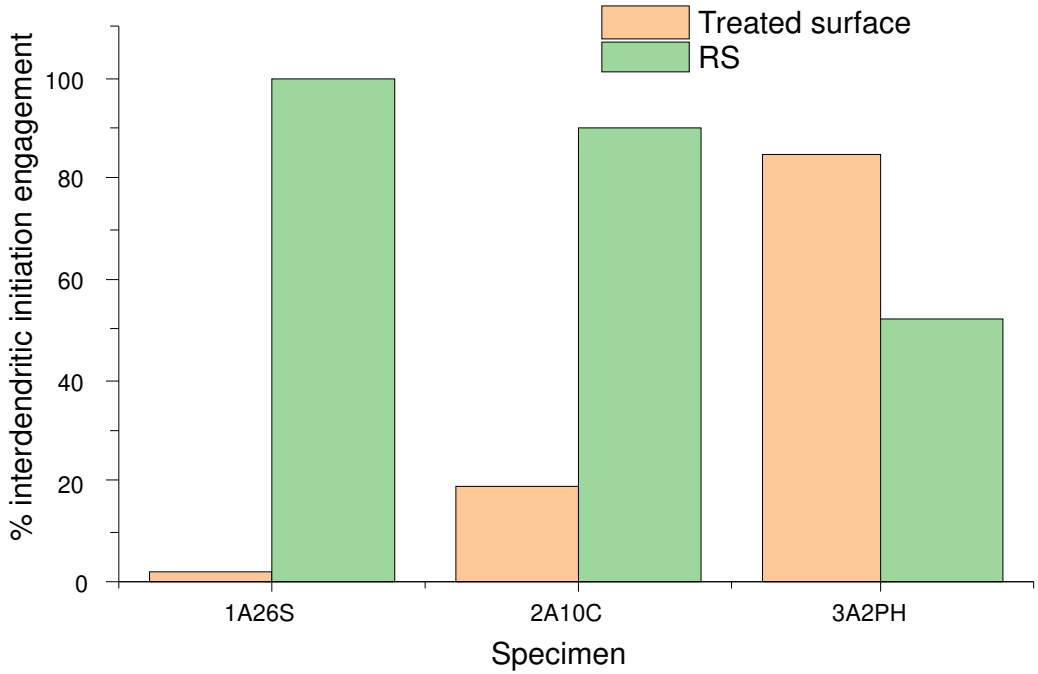
Based on the stress threshold values, the ranking of resistance in PWR conditions seems to be:
SAM > STI > RS



1A26S	2A10C	3A2PH
STI: 2%	SAM: 19%	SP: 85%
RS: 100%	RS: 90%	RS: 52%

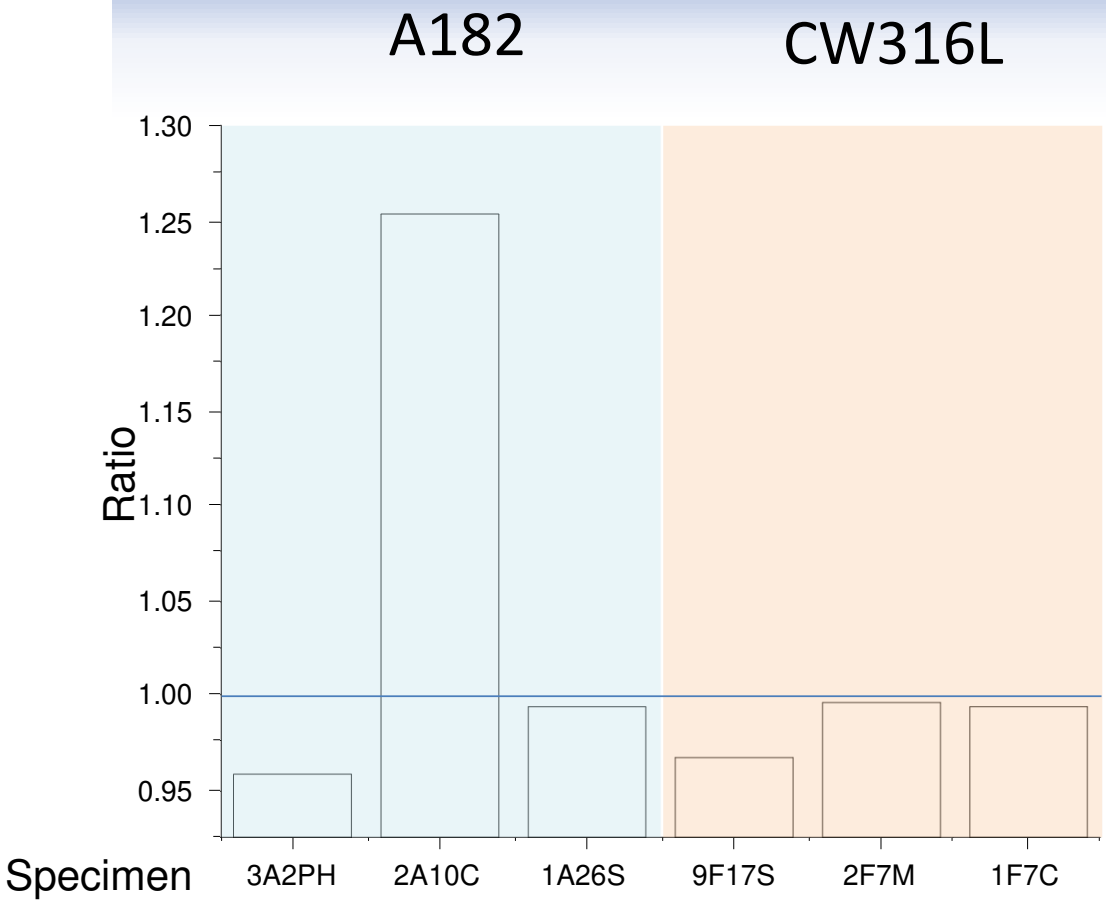
Interdendritic engagement at interface

% = [(a+b+c)/L]x100



Surfaces tested at some nominal strain rate ; $1x10^{-7}$ s^{-1}

$$\text{Ratio} = \frac{\sigma_{Machined\ (STI-SAM)}}{\sigma_{Reference(RS)}}$$



Relationship between the threshold values obtained for the different finishes compared to those obtained in the same specimen for the reference surface (RS). All specimens at the lower strain rates

Values < 1 shows a better performance of the RS side, while values > 1, better performance of the machined side.

CW316L

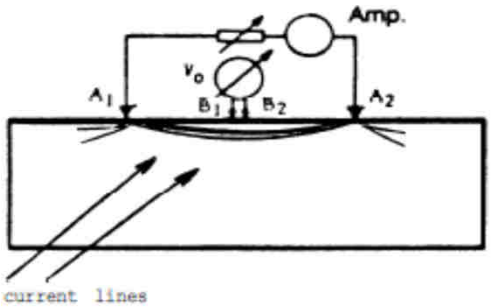
RS surface seems to have better behavior in all cases for CW316L.

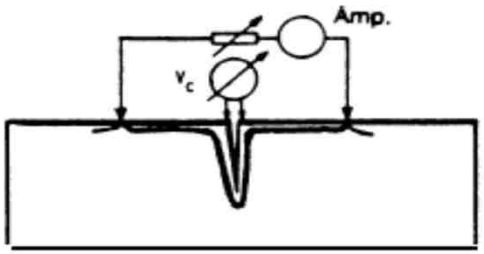
A 182

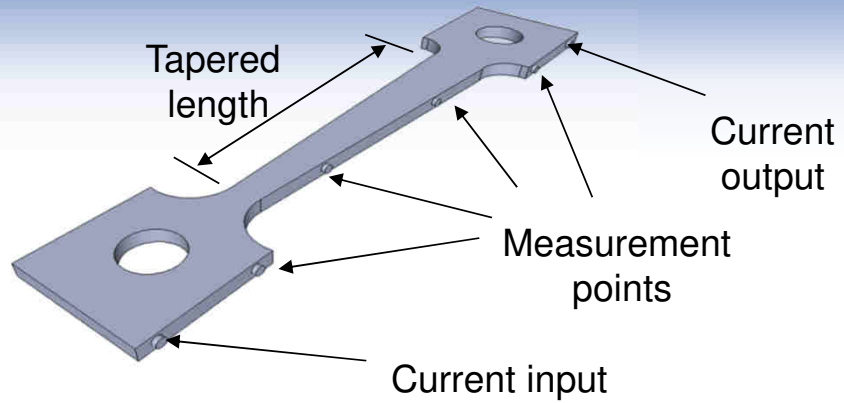
SAM surface shows some improvement with respect to the RS and STI finish for A 182. However, the fractography indicates the presence of interdendritic initiations not observed in STI.

DCPD monitoring

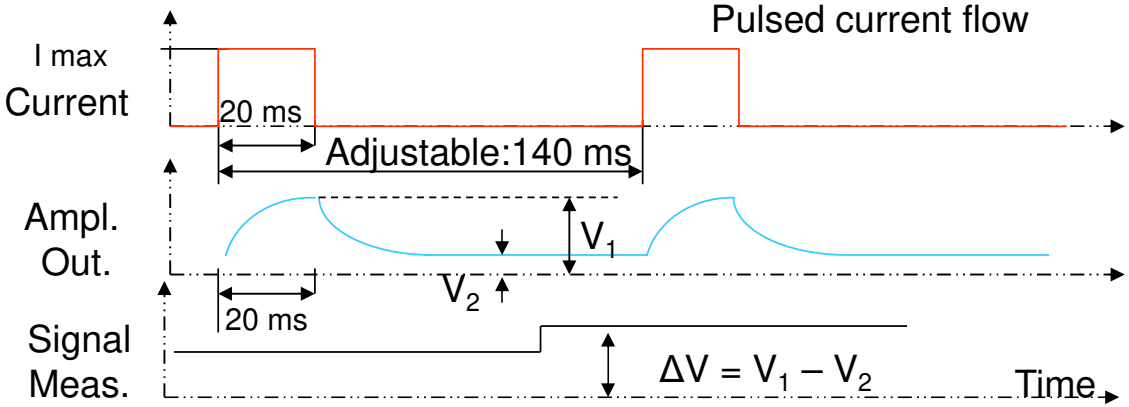
- Potential Drop Techniques
  - DCPD; ACPD

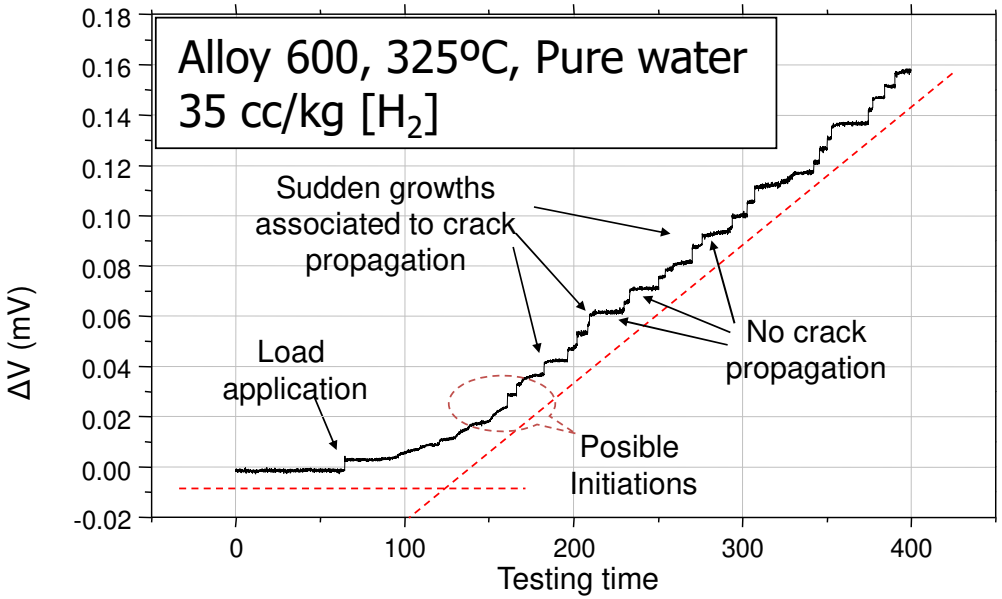




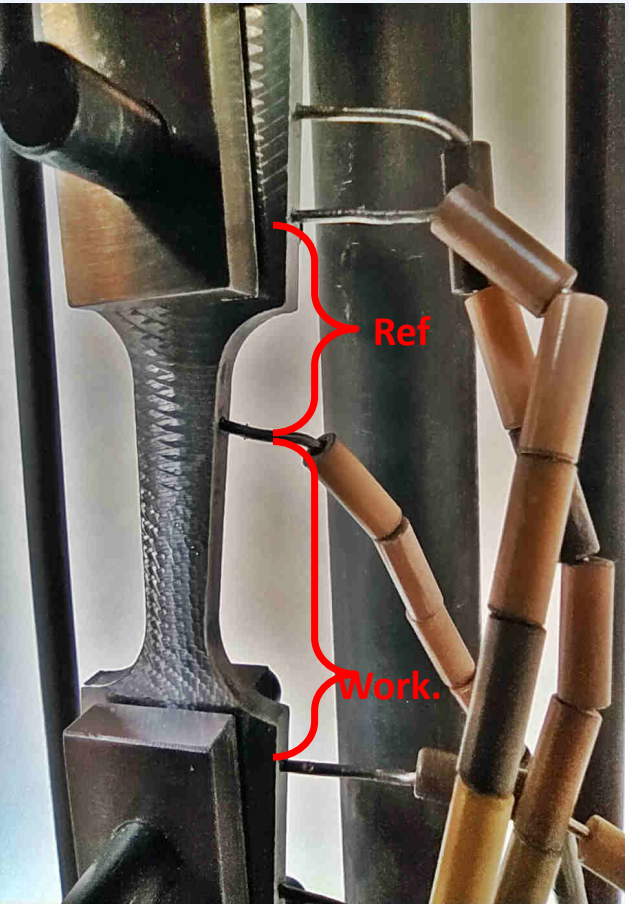


DCPD scheme



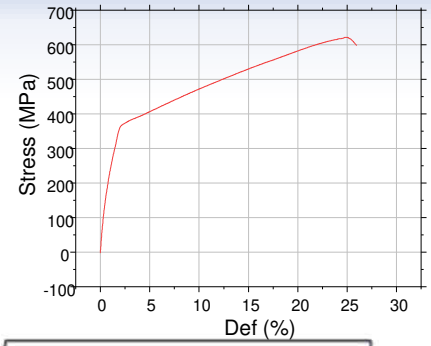


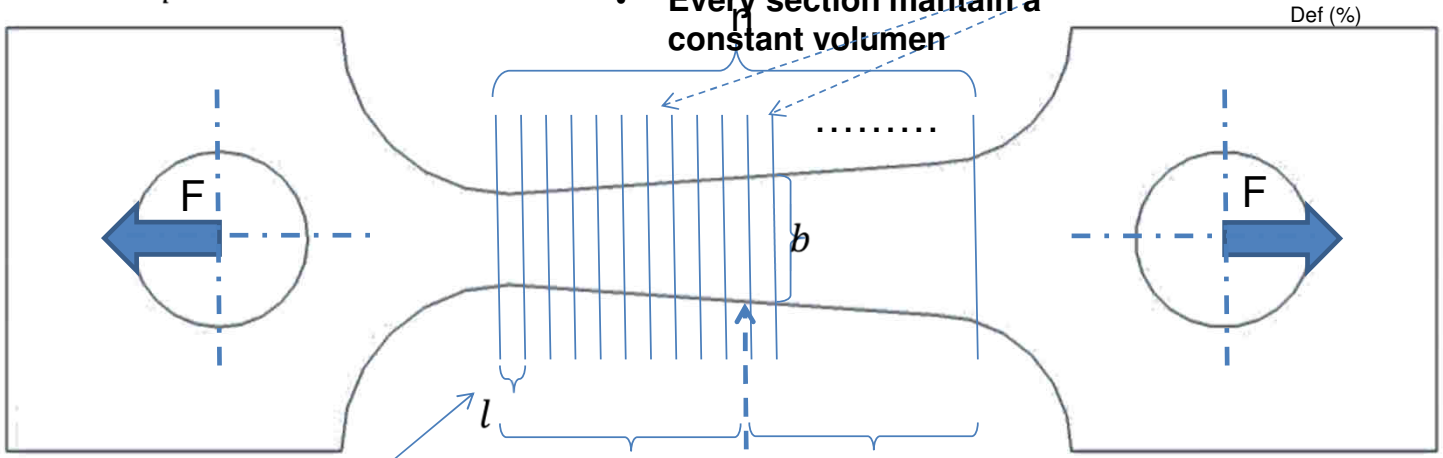
Position of PD electrodes attached to the specimen (measure & estimation of PD signal)



$$\frac{\Delta V_{Work} = \sum_{n=0}^{pos} \rho . \frac{l}{s}}{\Delta V_{Ref} = \sum_{n=pos}^{n} \rho . \frac{l}{s}} = Simulated\ DCPD$$

- **Every section is following the constitutive material law, from stress&strain curve at test temperature**
- **Every section mantain a constant volumen**





$$\Delta V = I \times R = I \times \frac{\rho\, l}{s}$$

$$S_n = en\ x\ bn$$

$$Volume_n = cte = S_n\ x\ l_n = e_n\ x\ b_n\ x\ l_n$$

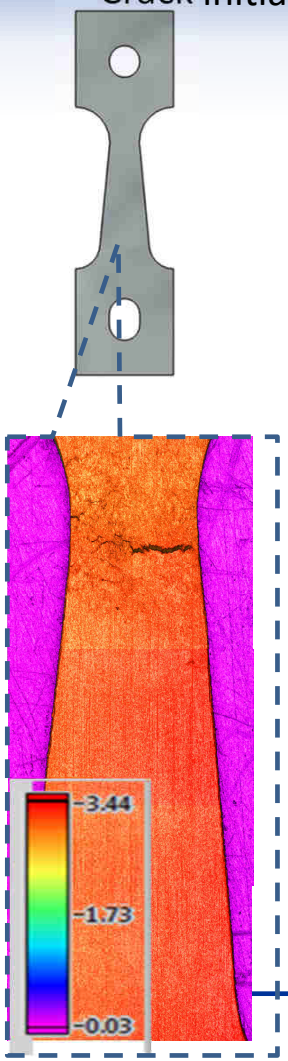
Working | Reference

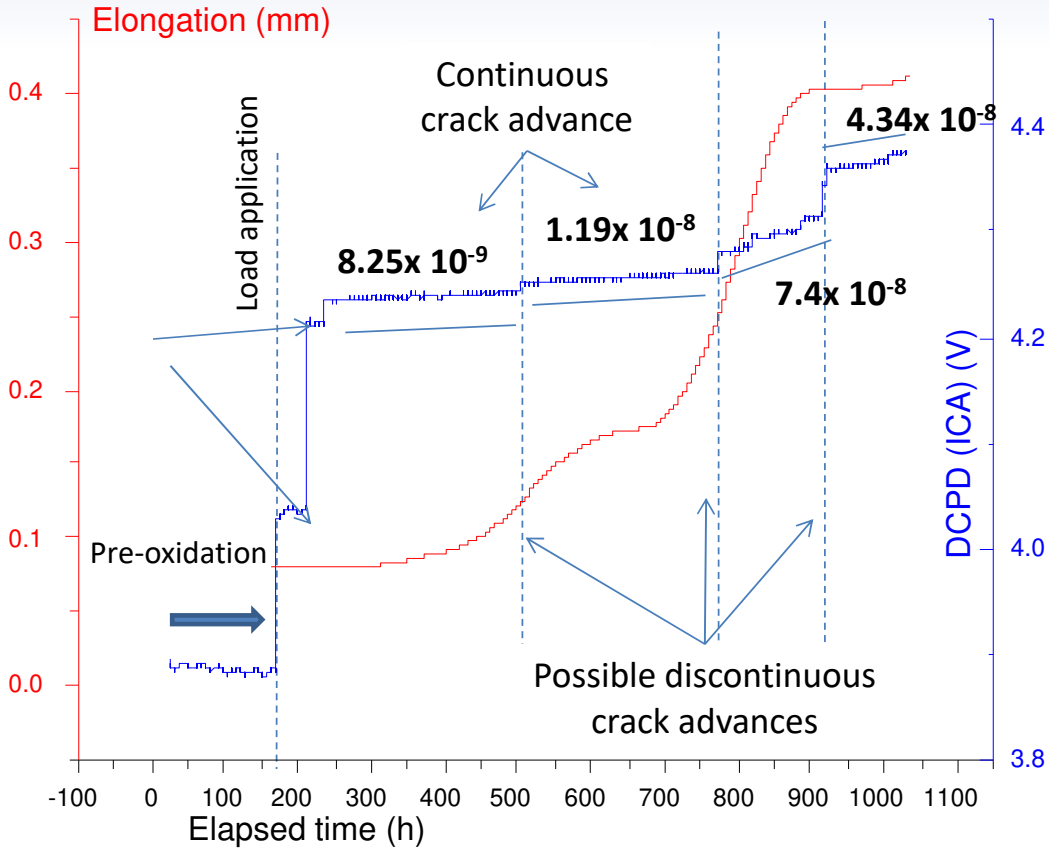
Pos. of welded electrode

$$e_n = thickness\ at\ n\ section$$

Monitoring constant load test by PD

Crack initiation/growth in a constant load test t≈1000 h (Alloy A 182)

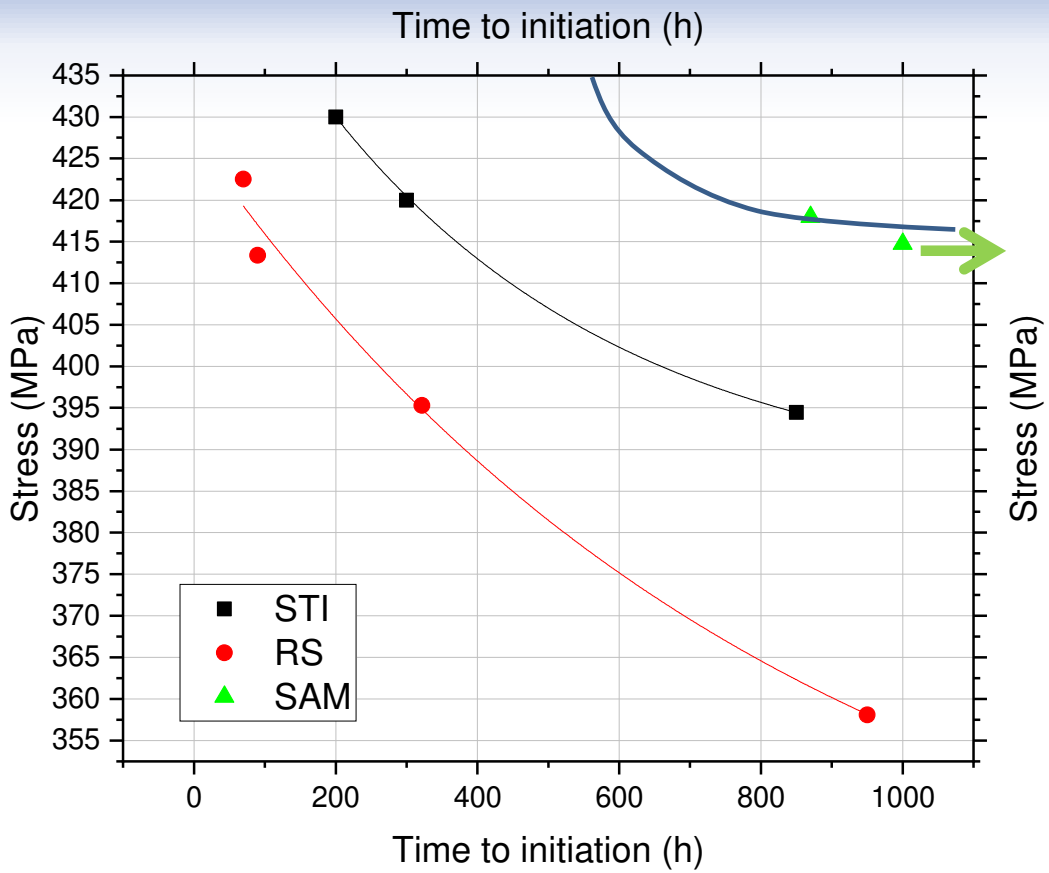




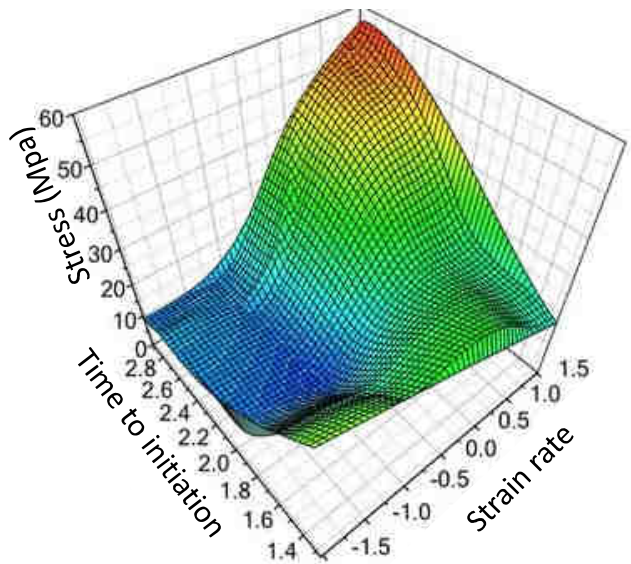
- Position of the last crack observed determine the "critical stress" at the strain rate level
- Time initiation determined by:
  - Scheduled shut downs and visual+ microscopic inspection
  - Monitoring by DCPD
    - Not always the signal is clearly identifiable with initiations
    - It is not possible to determine on which side the initiation/growth is occurring ➔post-mortem fractographic analysis
  - By elongation measure
    - Not applicable for several specimens in the same load chain

CGR estimated assuming a crack from minimum with section in specimen and/or fractography

Time to initiation from constan load test & CERT tests



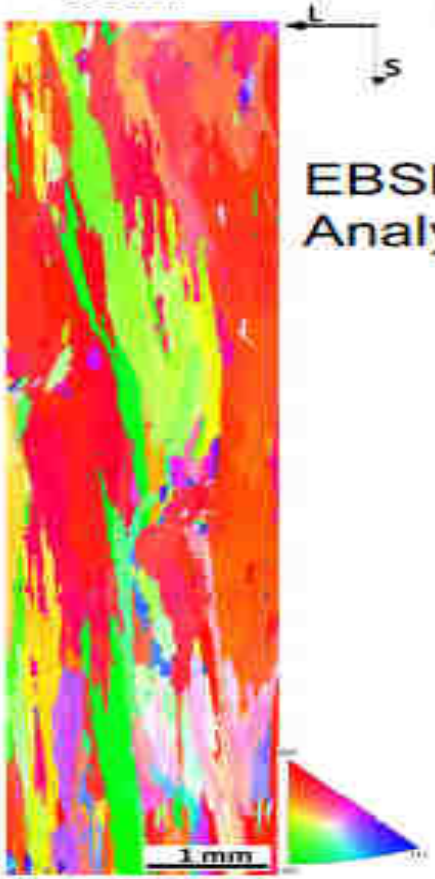
- Obtained mainly from the PD signal in constant load tests.
- With the PD signal, it is not possible to discern on which side the initiation is taking place, so the initiation time is an estimate based on the post-mortem analysis of the specimen.
- Diagrams Stress & time to initiacion and strain rate obtained from CERT tests



Alloy 182

TKD Characterization of UltraFine-Grained Layer: FIB Lift-out TEM Specimens (TS plane)

Machined "Crown"

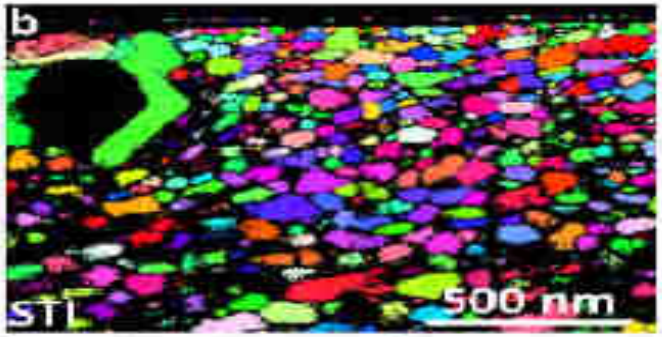


EBSD Analysis

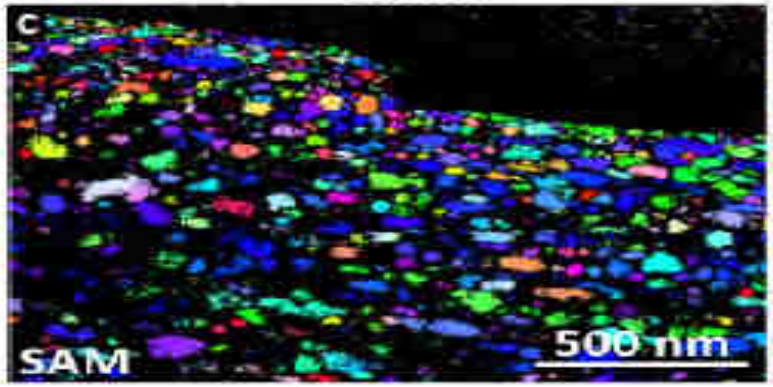
"Root"

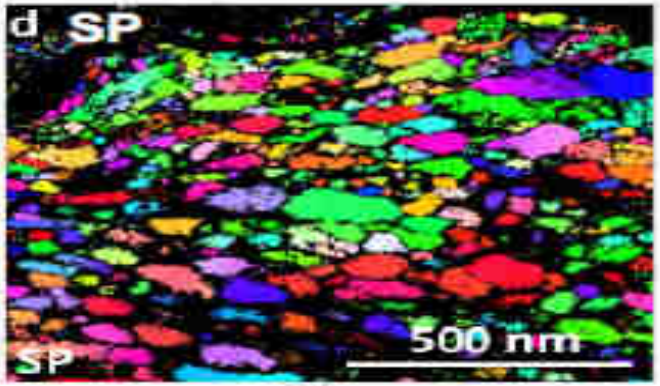
Reference

STI



SAM1

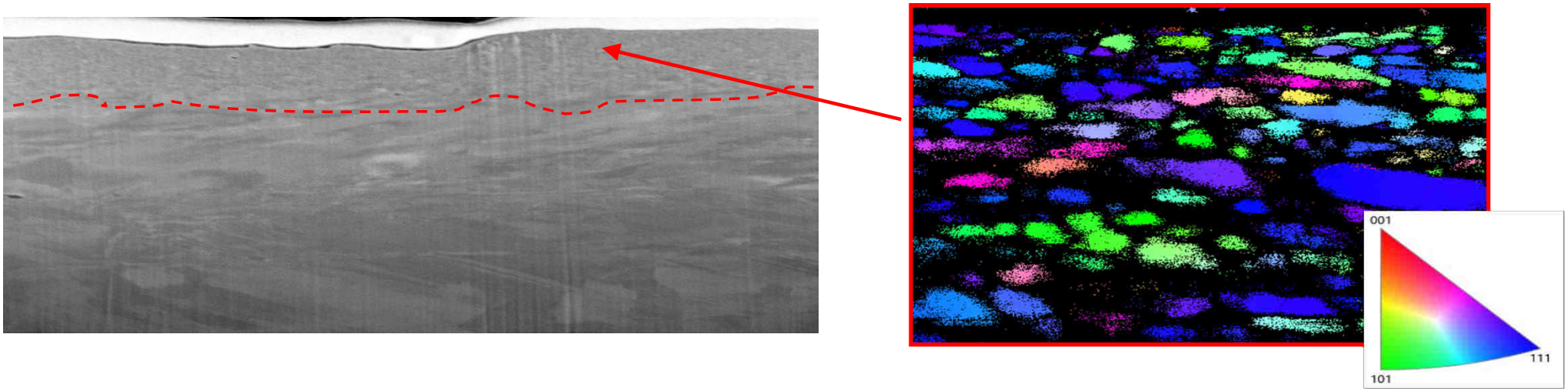




316 L

Surface-near surface

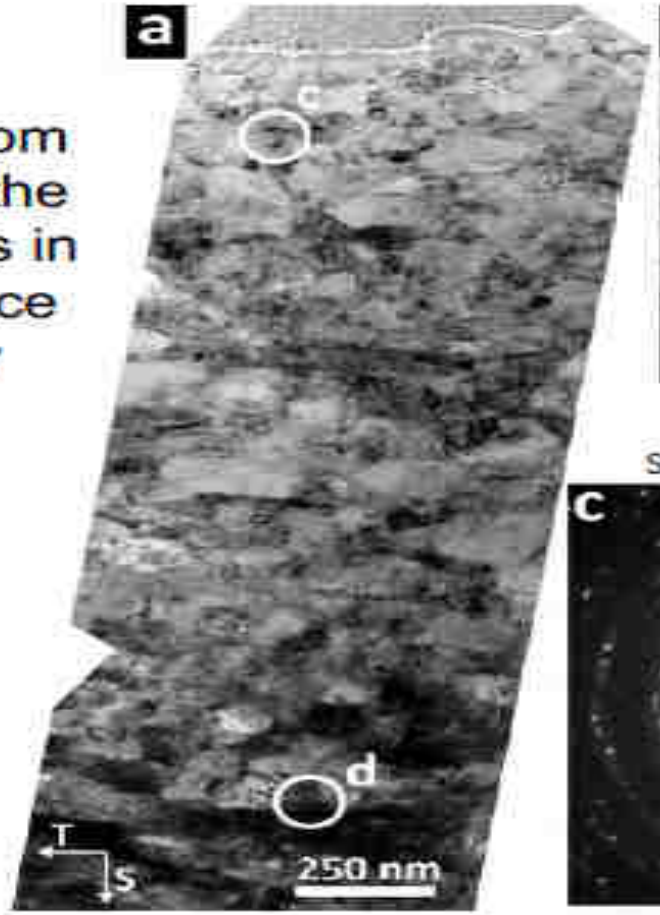
The ultrafine-grained layer (~2 µm thickness) associated with the STI treatment was characterized by grain sizes with dimension less than 300 nm.

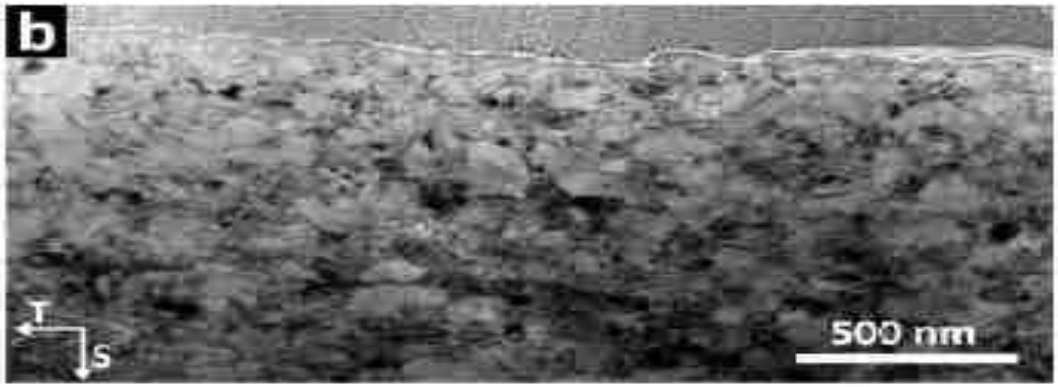


CW 316L Machined Tapered Specimens

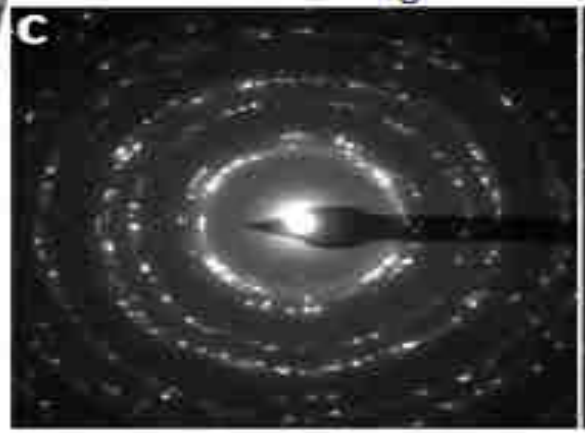
Cross-section TEM images of the SAM2 As-Machined Surface

Note the random orientation of the ultrafine grains in the near-surface region ("spotty ring" SADP).

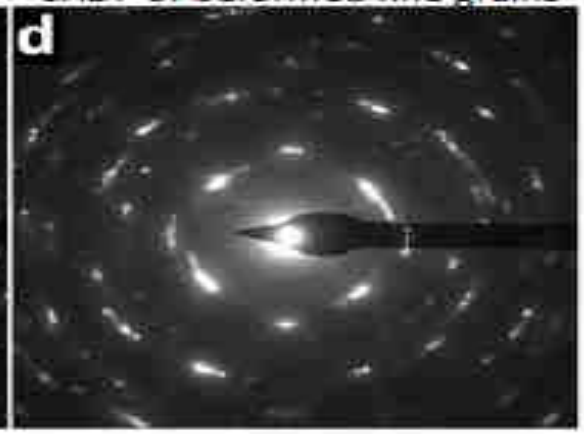




SADP of ultrafine grains



SADP of deformed fine grains



Results:

- Looking at stress threshold for EAC crack initiation SAM finish seems to improve the performance of the traditional machining and the manual polished finish, which presents the worst performance of the three surfaces.
  - Resistance of surface to EAC: SAM> STI> RS
  - However, the fractography indicates some isolated areas of interdendritic initiation sites, which have not been observed in traditional machining ➔ worse crack growth behaviour.
- The microstructural analyses carried out in the project indicate the presence of nanostructured layers of different depths, undoubtedly key in terms of their resistance to cracking.
  - Oxide formation and stability
  - Intergranular crack initiation time shorter, but transition from shot cracks to a unique crack growing may be arrest by other nanograins.
- For CW316L and for the applied cold work level (approx 13%) there is no clear benefit in the applied surface treatments. However, this material is itself very resistant, so the degree of cold working may not have been sufficient.
- The surfaces treated with peening are not directly comparable, since the high surface plastic deformation (which confers the compressive stress state) is very sensitive to deformation, so that after a small deformation the stress state can be reversed. Only can be compared in constan load tests
- All machining processes used have produced ultrafine-grained layers or different thickness in both materials, although it is not clear enough yet that they have a beneficial role in increasing resistance to cracking.
- Accelerate test too much can lead to error in the estimation of the stress threshold.
- Although the original idea of testing two surface finishes on the same specimen seems a good idea, it has been observed that the weaker side affects the behavior of the other.

Thank you for your attention

For any query:

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Alberto Sáez-Maderuelo: alberto.saez@ciemat.es