

Appendix 1

List of participants and excused persons

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Excuses received for the EFC WP15 meeting 8-9th March 2004 Milano

Appendix 2

EFC WP15 Activities

EFC Working Party 15 « Corrosion in Refinery »Activities

The following are the main areas being pursued by the Working Party:

Information Exchange

- Sharing of refinery materials /corrosion experiences by operating company representatives.

Forum for Technology

- Sharing materials/ corrosion/ protection/ monitoring information by providers

Publications

- Survey on amine unit corrosion
- Prevention of corrosion by cooling waters
- Items based on papers from previous conferences / meetings plus invited contributions
- Publications of the papers of the joint Workshop between WP 15 and others WP (e.g. 2004 Workshop "corrosion by hot gases and combustion" with WP3).

WP 15 meeting March 9th 2004

EFC Working Party 15 « Corrosion in Refinery »Activities

WP Meetings

·One WP 15 working party meeting in Spring,
(late one on 10th April 2003 in Pernis (NL)

·One meeting at Eurocorr in conjunction with conference,

Eurocorr Conference sessions (September)

WP15 page in EFC Web site <http://www.efcweb.org>

WP 15 meeting March 9th 2004

EUROCORR 2004

European Corrosion Conference

Long Term Prediction & Modelling of Corrosion

12 - 16 September 2004

Palais des Congrès Acropolis

NICE, France

European Federation of Corrosion
EFC event No. 266



Sessions and workshops

Workshops (indicated in green) deal with topics that are complementary to or more specific than the topics of the Working Parties.

- 1- Corrosion and Scale Inhibitors in Industrial Environments
(organisers: G. Schmitt, F. Moran)
- 2- Local Probe Techniques for Corrosion Research
(organisers: R. Oliva, R. Akid, V. Maurice, Ph. Marcus)
- 3- Corrosion by Hot Gases and Combustion Products with Emphasis on High-Temperature Corrosion in the Chemical and Petrochemical Industries
(organisers: M. Schütze, F. Ropital)
- 4- Prediction of Long Term Corrosion Behaviour in Nuclear Waste Systems (organisers: D. Crussel, D. Féron, J.-M. Grass, D.D. Macdonald)
- 5- Environment Sensitive Fracture, Corrosion Deformation Interactions
(organisers: J.-M. Olive, J.-M. Boursier, D. Delafosse)
- 6- Surface Science and Mechanisms of Corrosion and Protection (organiser: Ph. Marcus)
- 7- Corrosion Education and Computer Applications
(organiser: J.M. Costa)
- 8- Physico-Chemical Methods of Corrosion Testing
(organiser: J.H.W. de Wit)
- 9- Marine Corrosion (organiser: B. Espelid)
- 10- Biofilms and Materials in Natural Environments
(organisers: R. Gubaer, B. Tribollet)
- 11- Corrosion of Steel in Concrete
(organisers: J. Mietz, G. Tacke)
- 12- Marine Corrosion Workshop on Copper and Aluminium Alloys in Seawater
(organisers: B. Espelid, D. Féron, G. Ferrari, M. Jasner)
- 13- Corrosion in Oil and Gas Production (organiser: S. Olsen)
- 14- Coatings (organiser: L. Fedrizzi)
- 15- Corrosion in the Refinery Industry (organiser: F. Ropital)
- 16- Cathodic Protection (organiser: M. Roche)
- 17- Corrosion in the Automotive Industry
(organiser: F. Blöckenhörn)
- 18- Tribocorrosion: Industrial Needs and Scientific Insights
(organisers: J.-P. Célis, P. Pontbiaux, S. Mischler, M. Stack)
- 19- Heritage Conservation / Corrosion of Archaeological Objects (organisers: Ph. Dillmann, G. Béranger)
- 20- Non Destructive Techniques and Corrosion Monitoring
(organiser: Ph. Berge)

<http://www.scifrance.org/congres/eurocorr2004>

WP 15 meeting March 9th 2004

<http://www.efcweb.org/>

Publications

- EFC Guideline n°40 « Prevention of corrosion by cooling waters » (available soon)
- EFC Guideline n° 42 Collection of selected papers (in charge by John Harston)
- EFC Guideline on corrosion in amine units
- Future publications
 - EFC Guideline on Corrosion Under insulation
 - Collection of papers of Eurocorr 2004 joint session WP 3 + 15
 - other suggestions ?

WP 15 meeting March 9th 2004

Appendix 3

Review of Literature

Hennie de Bruyn Statoil

EFC WP15 - Corrosion Literature

Eichier Edition Affichage Insertion Format Eregistrements Outils Fenêtre ?

Refinery Corrosion Literature

EFC WP15 - REFINERY CORROSION LITERATURE

Main Groups

PROCESS CORROSION

ENVIRONMENTAL CRACKING

EXTERNAL CORROSION

Specific Corrosion Mechanisms

Amine corrosion

High temperature H₂S/H₂ corrosion

High temperature hydrogen attack (HTHA)

High temperature oxidation

High temperature sulphidic and naphthenic acid corrosion

Hydrochloric acid (HCl) corrosion

Hydrofluoric acid (HF) corrosion

Sour water corrosion

Sulphuric acid (H₂SO₄) corrosion

Literature

Author
Blount, A.L.

Title
"Corrosion by Naphthenic Acids"

Reference
API Proceedings vol. 11, no. 75, p. 102 (1930).


Abstract
The work refers the corrosion that affected the furnace tubes, the transfer line and the overhead condensers of a lube and asphalt unit, the test carried out to explain them as due to naphthenic acid attack and to the high velocity, the consequences of the mitigating actions (injection of solution of CaO and of caustic) and the results of tests made on alloy materials (the Alloy 16-18Cr-8-10Ni has a better resistance than the alloys 75Cu-20Ni-4.8Sn and 70Cu-29Zn-15Sn).

Year of publication: 1930 Filing reference:

Print this list

Entr: 1 sur 169

Written and developed by:
[H.J. de Bruyn - Statoil A.S.A](#)
(March 2004)



Mode Formulaire

EFC WP15 - Refinery Corrosion Literature

Main group: **PROCESS CORROSION**

Mechanism: **High temperature H₂S/H₂ corrosion**

NACE, "High Temperature Hydrogen Sulfide Corrosion of Stainless Steel", NACE Technical Committee Report, Corrosion, January 1968, pp. 271 - 311.

NACE, "Iso-Corrosion Rate Curves for High Temperature Hydrogen-Hydrogen Sulfide", NACE Technical Committee Report, Corrosion, Vol. 15, March 1959.

Cooper, A.S. & Dawidk, A., "High Temperature Corrosion by Catalytically Formed Hydrogen Sulfide", Corrosion, vol. 18 (p. 8), 1962, p. 2911 - 2981.

McCormy H.F., "High Temperature Sulphidic Corrosion in Hydrogen-Free Environment", Proc. API, vol. 43 (II), 1963, p. 78 - 96.

Cooper, A.S., "High Temperature Mercaptan Corrosion of Steel", Corrosion, vol. 19 (p. 11), 1963, p. 3961 - 4011.

Stranford K.N., "The Sulfidation of Metals and Alloys", Metall. Rev., vol. 138, 1969.

Cooper, A.S. & Goman, J.W., "Computer Correlations to Estimate High Temperature H₂S Corrosion in Refinery Streams", Mater. Prol. Perform., vol. 10 (p. 1), 1971, p. 31 - 37.

Forrest, Z.A., "High Temperature Degradation of Structural Materials in Environments Encountered in the Petroleum and Petrochemical Industries: Some Mechanistic Observations", Anti-Corrosion, vol. 32 (p. 11), 1985, p. 4 - 9.

Griffith, J., "High Temperature Sulphidic Corrosion of Steel", Process Industries Corrosion - The Theory and Practice, NACE International, 1986, p. 367 - 372.

Griffith, J., Merrick, R.D. & Schaefer, L.R., "Corrosion in Petroleum Refining and Petrochemical Operations", Metals Handbook, vol. 13, ASM International (1989) 1262 - 1287.

Appendix 4.1

Corrosion Under Insulation

Fixed equipment preservation

Andrew Kettle Chevron Texaco

Fixed equipment preservation

Vision

"A safe plant with world class levels of availability maintained at optimum cost"

Fixed equipment preservation is a major factor in achieving our Reliability/Maintenance vision for Pembroke Plant

Fixed equipment preservation

Pembroke Status in mid 2003

- Leaks due to external corrosion = 25 / annum
- IAN's due to external corrosion = 75 / annum
- LPO associated with external corrosion over £2.5MM / annum
- Furmanite clamps for external corrosion £250K / annum
- Line replacement / painting costs due to external corrosion £1MM otr / annum, £2.5MM / annum I&T. Total £3.5MM / annum
- Design ETQ's £1.5MM / annum. Inspection £30K / annum.

Fixed equipment preservation

Total Cost to Pembroke

£ 7.78MM/annum

10% ROCE = \$7.0MM / Month

Reduction of Maintenance costs by \$6 to 7MM in 2004 to reach 1Q Solomons

Fixed equipment preservation

Historical Approach

Reactive ↔ Proactive

Incident / Inspection Driven

Difficult to manage and track performance

Fixed equipment preservation

FCCU Wet Gas Compressor



This short section of line was insulated for PP. The line is at ~ 60 °C.



LPO = ? Fortunately didn't delay startup.
Risk = Significant levels of H₂S in this line .

Fixed equipment preservation

Platformer Instrument Lines



Serious external corrosion in an area which failed to register of the CUI Radar screen.

- Classified as too hot on P&ID
- Fell between Inspection and Instrument responsibility

LPO = £700,000
Risk = Small Fire Occurred but could have been much bigger - look at metal loss on process side of first isolation



Fixed equipment preservation

It's not just Insulated lines

1-WC-604 Cooling water line which has suffered external paint breakdown and corrosion resulting in a leak.



LPO = Small but could have been big
Risk = Imagine if it wasn't water !!!
Even so a failure of this type could shut a unit down.

ChevronTexaco

7

Fixed equipment preservation

Operations and Maintenance

Joint Opportunity statement

"Minimise the Safety and Lost Profit Opportunity risks associated with external corrosion mechanisms through the definition and execution of a cost effective, proactive preservation program"

ChevronTexaco

8

Fixed equipment preservation

CPDEP Phase II - Selected Approach

- Geographic basis - prioritised and based on overall plant risk - service and business criticality
- One Team - One Budget ~ £ 4.5MM
(2.5 OPEX / 2 CAPEX)
- Close co-operation between ALL involved

ChevronTexaco

9

Fixed equipment preservation

EPP Team

Project Engineer - Dave Thomas
Civil Co-ordinator - John Skone
Painting and Insulation Inspector - Myles Doyle
NDT technicians - CAN Dedicated

Assignment Roles

Contract Strategy - Richard Brookes
Inspection Engineer - Area Engineer for Unit
Ops/Process Engineers - Area Engineer for Unit

ChevronTexaco

10

Fixed equipment preservation

The Prize

- Conducive to economic execution
- Ease of set up, tracking and administration
- Inspection Economies due to alteration of WSE - visual and external corrosion NDT programs
- Maintenance Economies due to integration of OTR program and collective repair strategy on a zone basis
- Easier to control for Operations - Less Interfaces

ChevronTexaco

11

Fixed equipment preservation

The Plan

- Optimum duration - 10 year rolling program
 - High risk plants first by Level 1 risk assessment - Set before I&T
 - 80% of risk carried by 20% of plant
- Inspection regime shifts with time
 - High risk items in other areas not left beyond normal inspection interval

ChevronTexaco

12

Fixed equipment preservation

What is happening now

- Risk assessment of Refinery Plants/Areas on consequence & business risk to develop plant phasing within 10 year program.
- Develop contract and execution strategy
- Zone selected plants based on logical execution and risk
- Develop work scope for zoned areas

Fixed equipment preservation

Zoning and Work Scope Definition

- P&ID reviews to set Equipment and Piping category
- Site surveys to confirm status
- Process and instrument reviews to assess need for re insulation
- Operations review to assess acceptability of zoning plan
- Technology reviews to assess suitable coating and insulation alternatives

Fixed equipment preservation

Working together

We need to swing the balance

Reactive ↔ Proactive

Fixed equipment preservation

Working together

We need to swing the balance - it will take time !

Reactive ↔ Proactive

Fixed equipment preservation

We already have good practices



A typical example of PP cladding applied on ULSG.

Perforated sheet and no insulation.

Safety Maintained
Integrity Maintained

IFO Goal achieved

Appendix 4.2

Corrosion Under Insulation

It's there. How do we manage it ?

Maarten Lorenz Shell Global Solutions

Corrosion Under Insulation It's there - how do we manage it ?

Many elements of the CUI issue are available,
but who has the overall view?

Building an EFC CUI guideline based on
information we did not realise we already
have...

Presented by:

Maarten Lorenz

Material & Inspection Technology
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What is the problem with CUI?

- **It is not visible**
(covered by insulation)
- **It may start any time**
(depending on conditions)
- **It is largely impossible to predict where it occurs**
(vapour travels through insulated system)
- **It is generally difficult to assign a corrosion rate**
(humidity may not be constant)
- **It costs a lot of money to manage**
(maintenance, inspection, failures)
- **Ownership is not clear**
(who is dealing with it)

Costs associated with CUI

- Costs of maintenance of insulated systems
- Costs of inspection for CUI
- Costs of failures caused by CUI

Improve CUI management

- Elimination
...through removal of insulation
- Prevention
...through better design (coatings, insulation systems)
- Mitigation
...through maintenance and inspection

Efforts based on:

- risk assessment
- cost/benefit ratio

Elimination of CUI

- Cages for personnel protection
- Challenge value of energy savings
- Evaluate alternatives to insulation
- Change type of steel

Prevention of CUI

- Paints and coatings
 - Types of paints/coatings
 - Quality of application
- Insulation material
- Reduce water ingress
- Ventilation
 - Stand-off insulation systems
- Reduce water vapour travel through system
 - Isolate insulated systems (corrosion loops)
- Drains

Mitigation of CUI

- Avoid mistreatment of insulated systems
- Adequate maintenance of insulated systems
- Inspection for CUI
- Monitoring of water presence
- Monitoring of process conditions (temperature!)

Maintenance of insulated systems

- Risk-based schedule
- Focus on coating and prevention of water ingress
- Check for presence of water (vapour)

Inspection of insulated systems

- Risk-based schedule
- Intrusive: removal of insulation
- Non-intrusive: reliable inspection techniques?

Monitoring water presence & temperature

- No CUI without water
- Value of thermography inspection
- Detect water presence
- Monitor temperature to determine susceptibility to CUI

What can we do about the problem of CUI?

- **It is not visible**
 - removal of insulation
 - apply reliable non-intrusive inspection technique
- **It may start any time**
 - Monitor water presence, coating condition & temperature
- **It is largely impossible to predict where it occurs**
 - Isolate insulated systems (corrosion loops)
- **It is generally difficult to assign a corrosion rate**
 - Monitor water presence and temperature
- **It costs a lot of money to manage**
 - Risk-based assessment to optimise mtce/insp schedule
- **Ownership is not clear**
 - All disciplines have to be involved

Appendix 5

Optimised inspection planning

Maarten Lorenz Shell Global Solutions



Shell Global Solutions

Optimised Inspection Planning is more than Risk-Based Inspection

What assistance would you like to have with optimising your risk-based inspection schedule?

Presented by:
Maarten Lorenz

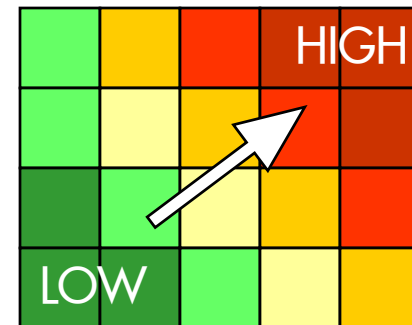
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Risk-Based Inspection - Benefits

Risk-Based Inspection (RBI) provides for equipment:

- Identification of degradation mechanisms and localisation
- Indication of potential risk level
- Prioritisation of inspection effort
- Inspection intervals
- Suitable inspection method



⇒ **RBI has to lead to:**

- More efficient inspection programme
- Higher plant reliability & availability
- Increased plant safety

DOES IT?

Risk-Based Inspection - the way forward

RBI ⇒ high level inspection plan

Increased plant availability & reliability

⇒ more and optimized in-service (non-intrusive) inspections.

⇒ optimize conventional NII (FFP, POD, coverage)

In this respect, **RBI needs to be followed up** by determining:



- ... if non-intrusive inspection has a potential benefit
- ... which non-intrusive inspection techniques can be used
- ... what critical defect can be found by inspection
- ... what the inspection coverage shall be
- ... what the inspection effectiveness is
- ... what the reliability of inspection findings is

The need for optimised inspection planning

A detailed inspection plan needs to be designed, which aims at:

- **Reducing intrusive inspections**, leading to shorter shutdowns and longer run length (improved plant availability)
- **Improved confidence** of conventional NII

while at the same time it is:

- **Technically feasible**
 - can it find the defect you are looking for?
- **Cost-effective**
 - is change to non-intrusive inspection worth the effort?

⇒ **What do you need to do this?**

Support for shift to Non-Intrusive Inspections

- **Identification of opportunities** for non-intrusive inspections in refineries as an alternative to intrusive inspections?
- Assistance in determination of **coverage**?
- Assistance in **choice of technique**?
- Assessment of **confidence** in results?
- Role of wall thickness or corrosion **monitoring**?
- Assessment of **critical defect** with respect to the integrity of the structure?
- **Risk reduction** as a result of inspection effort?

What is Shell-Non Intrusive Inspection (S-NII)?

- S-NII is a structured methodology for the detailed design of non-intrusive inspections
- It is usually applied within a framework of Risk based Inspection planning
- S-NII helps the user to determine:
 - Where to inspect
 - Which technique to use
 - The inspection sensitivity
 - The coverage
 - The reliability of technique
 - The tolerable risk level

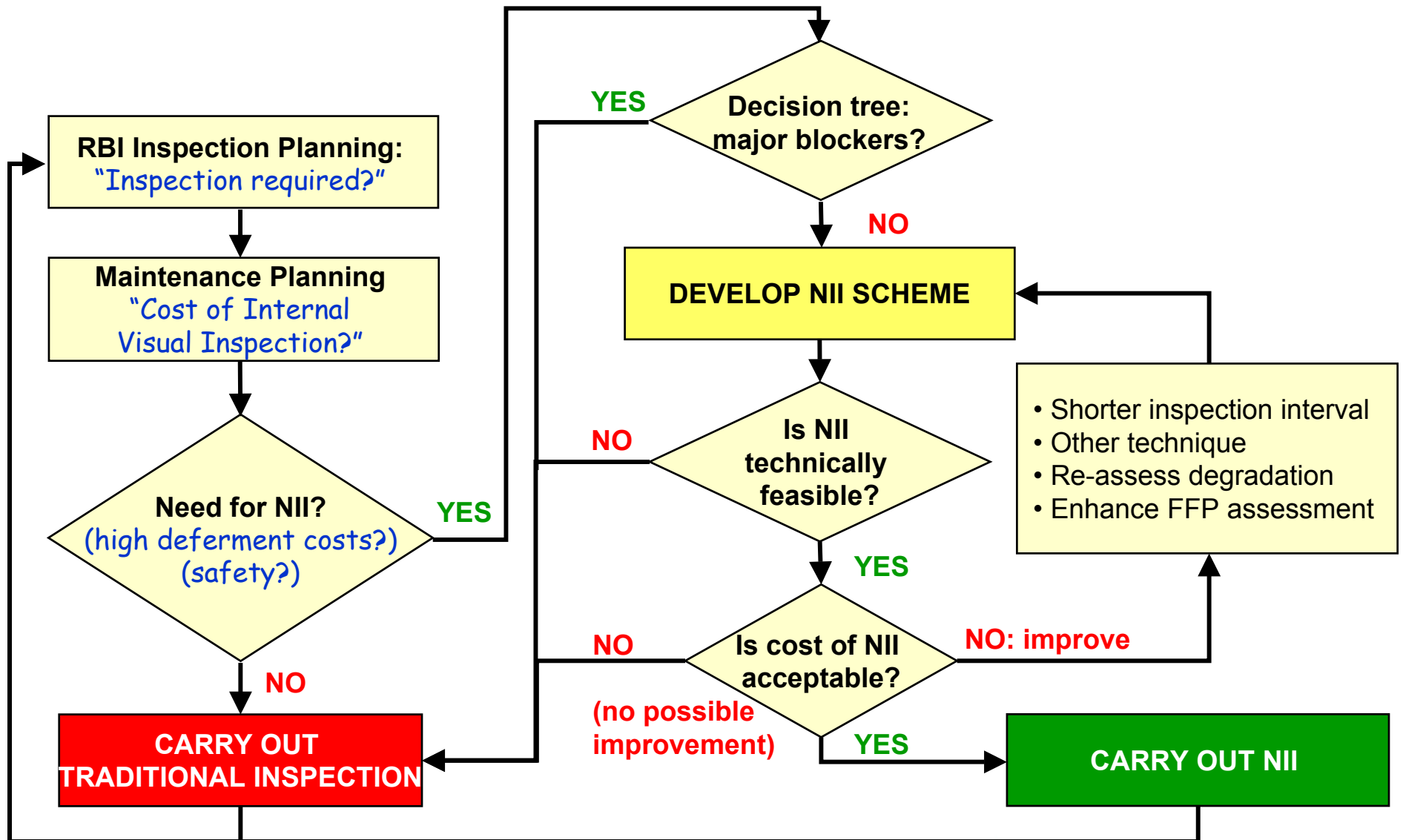


What are the benefits of S-NII?

- Replacing intrusive inspections by NII will lead to shorter shutdowns and longer runtimes, giving higher plant availability
- Additional benefits of using NII may come from:
 - Improved conventional NII leading to fewer unexpected failures
 - Reduced exposure of personnel to confined spaces
 - Reduction of hydrocarbon leaks (by not breaking flanges)
 - Reduced indirect cost of cleaning and waste disposal

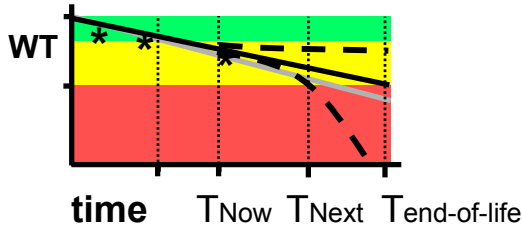


The process of Shell Non-Intrusive Inspection



S-NII integrates knowledge

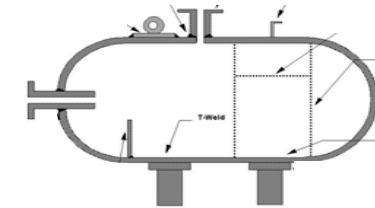
7. Update Remnant Life



S-RBI Criticality Matrix

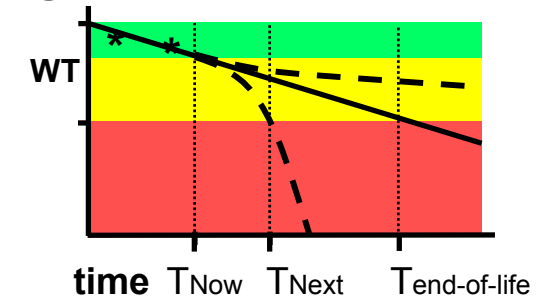
M	H	E	E
L	M	H	E
N	L	M	H
N	N	L	M

0. RBI-Inspection planning



1. Equipment components

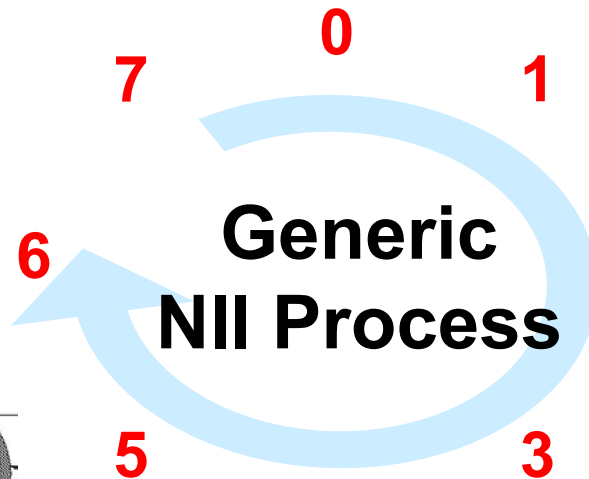
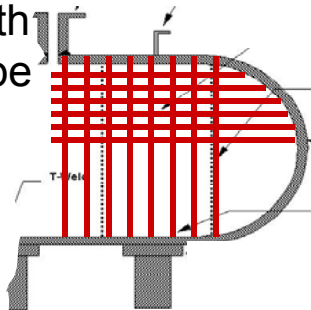
2. Characterize degradation mechanism



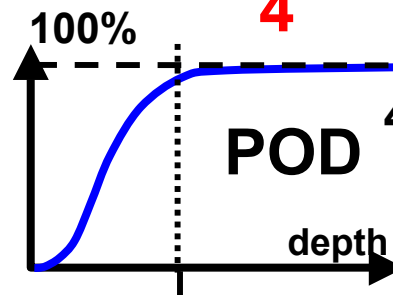
6. Analyse NDT data

5. Implement NII

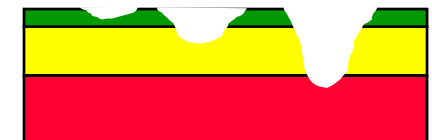
- comply with work scope



3. Define maximum allowable defects

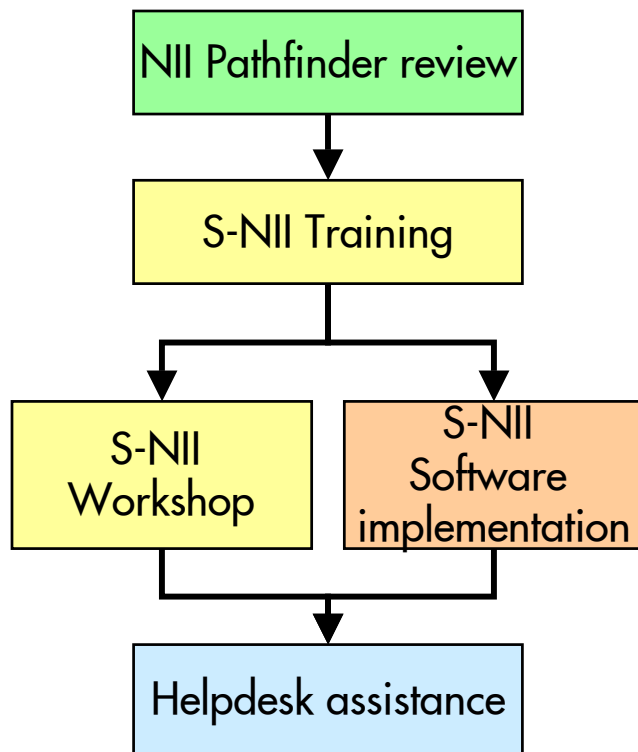


- ## 4. Nii design
- technique
 - confidence/cost



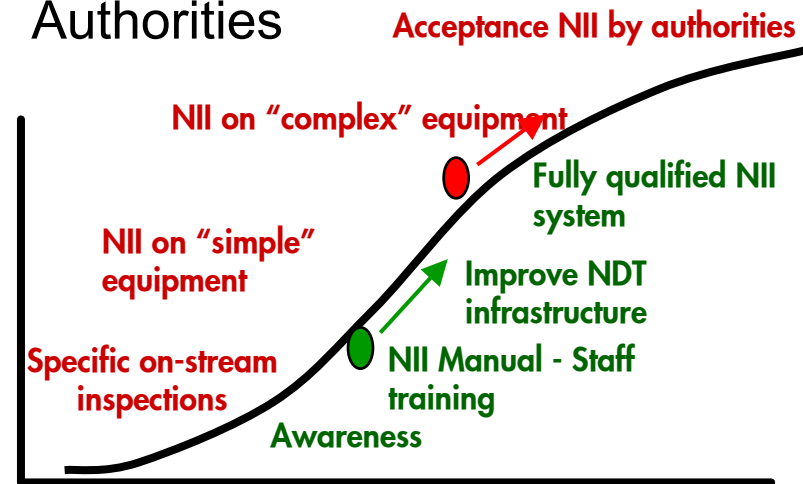
How is S-NII implemented?

training and implementation support programme:



S-NII implementation provides:

- Consistent and verifiable planning
- Enhanced inspector competence
- Knowledge at your fingertips
- A convincing case towards Authorities



Learning curve for NII Implementation

S-NII is supported by software

S_NII - [S_NII1.nii:1]

File Edit View Mode Configuration Window Help

UT Normal beam - Line scan

Tag	Asset class	Stratum	Stratum co	Shell materi	Commissio	Shell wall th	Shell corros	Design WT	Original CA	Base date	WT at base	CA at base	Corrosion r
ST48	Vessel	1	Vessel Weld	Steel	1-Jan-1990	20.0	3.0	17.0	3.0	1-Jan-1995	20.0	3.0	0.1
ST48	Vessel	2	Stratum comp	Steel	1-Jan-1990	20.0	3.0	17.0	3.0	1-Jan-1995	25.0	3.0	0.1

S-NII Steps

- Import Data
- Calculate Life Cycle
- Define Stratum
- Default Scheme
 - Gridding
 - Screening
 - Sampling
- Inspection Technique
- Cost Model

Define Stratum

Number: 1, Component: Vessel Weld

Component in Phase 1 Of Life cycle ?

Component in Phase 2 Of Life cycle ?

Component in Phase 3 Of Life cycle ?

Is sampling possible ?

Develop Gridding scheme

Develop Sampling scheme Samp-U

Develop Screening scheme Screen-U

Develop Sampling scheme Samp

Default Scheme | Gridding | Screening | Sampling | Technique | Cost Model

Input for Life Cycle Phase

Commissioned date: 1-Jan-1990
 Nominal WT: 20
 Corrosion allowance: 3
 Design WT: 17
 Design CA: 3
 Base date: 1-Jan-1995
 WT at base date: 20
 CA at base date: 3
 Actual corrosion rate: 0.1
 Confidence rating: HIGH
 Cons of Failure: MEDIUM
 Interval factor: 0.3
 Remnant Life: 30
 Next Insp Date: 1-Jan-2004

S-NII

Exceedance probability: 0.20
 Predictability: 4.0
 Average Corrosion Rate: 0.08 mm/y
 Stdev Corrosion Rate: 0.02
 Minimum Integrity Level: 1.0e-003

Remnant Life: 30.0 y
 Remnant Life Date: 1-Jan-2025
 Life cycle phase 1: 20-Jul-2015

Current Inspection Date: 1-Jan-2004
 WT (current RWT): 18.7 (1.10)
 Future Inspection Date: 20-Apr-2010
 WT (future RWT): 17.8 (1.05)

Apply Help

Remnant Life plot ST48

RL plot | Integrity Curve | RL gridding | RL screening | RL sampling

Integrity Curve and Minimum Integrity Level ST48

RL plot | Integrity Curve | RL gridding | RL screening | RL sampling

Appendix 6

Failure Case

PWHT of alloy clad vessels

Andrew Kettle Chevron Texaco

PWHT of Alloy Clad Vessels



ChevronTexaco

1

PWHT of Alloy Clad Vessels

Column Detail

- ASTM A516 Gr70 + 304L Grade S/S
- 50 mm plus 5 mm thickness

- Naphtha / Hydrogen and H₂S Environment
- 250 to 350 °C operating range

ChevronTexaco

2

PWHT of Alloy Clad Vessels

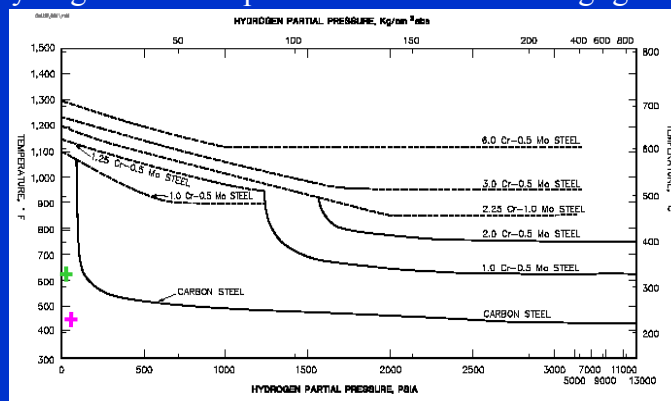
Specified Heat Treatment Schedule

- 3 fabricated sections
 - 593 to 677 °C for 2 hrs 15 minutes to comply with ASME code and Texaco GEMS.
- 2 closing weld seams
 - Localised post weld heat treatment at 593 to 677 °C for 2 hrs 15 minutes.

PWHT of Alloy Clad Vessels

Corrosion Potential

Hydrogen attack as per the Nelson curves - negligible.

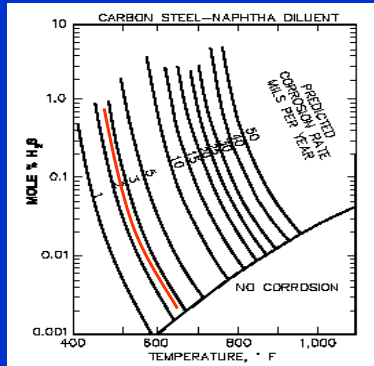


+ Bottom of Column + Top of Column

PWHT of Alloy Clad Vessels

Corrosion Potential

Hot Hydrogen / H₂S in Naphtha as per Couper-Gorman predicted at 2.5 mils per years (0.0625 mm per year)



ChevronTexaco

5

PWHT of Alloy Clad Vessels

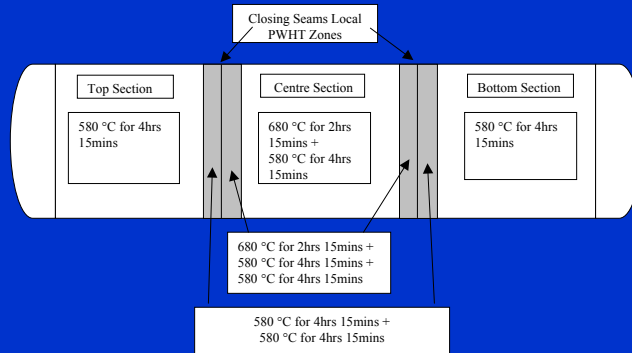


ChevronTexaco

6

PWHT of Alloy Clad Vessels

Mechanical reshaping and finished Heat Treatment condition



PWHT of Alloy Clad Vessels

- Cladding and Nozzle material tested for sensitisation
 - Cladding show some grain boundary carbide network
 - Nozzle material does not appear to be affected
- Susceptibility of 304 cladding to PASCC ?
- Susceptibility of 304 Nozzles to PASCC ?
- Operation and maintenance procedures under review NACE RP0170-97
- Q - Any success with ASTM G35-76. Difficulty appear to be in simulating polythionic acid conditions

Appendix 7

State of the art for portable diffractometers

X Ray Diffraction

G. Berti University of Pisa

PROPOSAL OF A NEW MOVABLE DIFFRACTOMETER FOR NDT APPLICATIONS IN SEVERAL INDUSTRIAL PURPOSES EITHER IN LABOARTORY OR IN FIELD¹

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Via S. Maria 53, 56126 Pisa, Italy

Most of the diffraction analysis are currently carried on inside of laboratories by using a number of different optional diffraction devices. Of these devices the specimen stage is one limiting the use of diffractometers when analysis are required to inspection large specimen or specimen with irregular shape. In general, this specimen stage is mechanically supported, it lies on a plane normal to the equatorial one and positioned on the goniometer axis. The main advantage of this configuration (typically the Bragg-Brentano configuration) is that it is the best known one and allows for precise calibration (i.e. computation of the non-removable systematic effects).

A new diffractometer has been designed, which tends to conserves as much as possible the advantages of the Bragg Brentano configuration; it removes the limits for the above mentioned applications, when collections of diffraction patterns are required for uses in fields (i.e. directly at the site of the piece to be investigated). In general in traditional XRD types of analyses the specimen is adapted to the best performance of the analysis. This limitation prevent XRD to be used as a strict NDT method. The challenge this new proposed device has the ambition to launch is to aggress the mentioned limitation by bringing XRD measurement directly on the industrial plants (possibly in services). The way to win the challenge might be not obvious; a small path has been already run and still the greatest is remain to reach the same performances as the traditional laboratory diffractometer.

The general goniometer configuration might be considered as a $\theta : \theta$ like arrangement, where the source and the detector are moved in the opposite directions. The general instrument might be considered as an industrial robot where the main arm is equipped by the main following devices:

1. A double Euler cardle supporting the diffraction devices and providing the diffraction measurements. The main Euler cardle contains the source, the detector and a number of diffraction optic devices can be mounted either for divergent and parallel beam applications. The radius of the main Euler cardle is about 20 cm. Source collimator and detector collimator can be properly arranged to minimize the radiation dispersion in the environment.
2. Movable devices providing three translational freedom degrees and one rotational degree. There are eight step motors providing all the movement for the fine positioning and control of the diffractometer performances.
3. Pointing devices and optical imaging detector (i.e. small lasers and camera) are also provided and the remote control software to perform the instrument positioning and the measurements. The remote control is based on programmable micro processors activating the eight step motors.

The following slides summarise the presentation by putting some emphasis on the role of XRD in the field of material diagnosis and characterisation. The presentation of the XRD movable equipment has been given by using a demonstration movie. Finally few preliminary results has been

¹ Work partially supported by ISPESL – Roma (research contracts 67/97,42/98). The work group is currently compose by:

C. De Petris, G. Augugliaro, P. Sofia (ISPESL)

L. Sani, F.De Marco, (University of Pisa)

S. Aldrighetti, F. Aldrighetti (OET)

Summary of the presentation given in EMI Tecnologie Research Centre - S.Donato Milanese for the EFC Working Party 15 Corrosion Refinery Industry Meeting - March 9,2004,


illustrated in the final slides, where data have been collected from monocrystalline silicon oriented specimen, powder of Alumina (Corundum) specimen, mechanically loaded. Residual stress across a welding cord ha been also investigated and the expected results are reported in the last slide.

PORTABLE DIFFRACTOMETER FOR NDT-X-RAY DIFFRACTION ANALYSES

Giovanni Berti - University of Pisa
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by the cooperation of
 ISPESL - CND Monteporzio Catone - Roma
 Officina Elettrotecnica di Tenno - Trento

Engineering and distribution by
XRD-Tools
 an Academic Spin Off of Univ. of Pisa xrd-tools@email.it



X-ray Diffraction (XRD)

X-Ray Diffraction is a physic process generated by the interaction of a x-ray beam with materials, (i.e powder crystalline lattices, metallic blocks, ceramics, thin layers etc.)

XRD is observed as a coherent scattering of waves from suitably ordered scattering points (atoms)

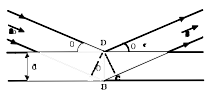
Bragg Law : $2 d \sin \Theta = \lambda$

d is the lattice parameter
 Θ is the detection angle
 λ is the beam wavelength

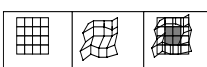
Bragg Law and Real Lattices

$2d \sin \Theta = \lambda$

B and D are two scattering points on two contiguous planes
 A and C are points determining the path difference of the beam scattering from the two points B and D



The Mean Equivalent Lattice
 Is a confined region of the real lattice, where compatible conditions exist with the Diffraction, when lattice deformations and systematic experimental contributions are taken into account.



$2(d+d') \sin(\Theta + \Theta') = \lambda + \lambda'$ Generalised Bragg Law

Potentialities and Objectives of XRD

Research&Development, Experiment and Technology

- Methods, Innovation, Instrumentation,
- **Standard Specimen, Standard Protocol and Good Practices**

Characterization, Diagnosis and Qualification

- **Physical properties, chemical properties, crystallography of materials**
- Qualification of mechanical properties of crystalline materials and non crystalline materials at the scale of nano-meters for specific applications and uses

Destructives and Non Destructives Tests

- Tests on manufatures, natural materials either in laboratory or in place
- **Test on industrial implants or for industrial processes.**

Professional Training

- Interdisciplinary: Mineralogy, Crystallo-chemistry, Agronomy, Biomaterials, Science and Engineering of Materials and Nano-materials,
- Engineering and Robotics, Metrology, Physics, Computer Sciences.)

Others Aspects (still not yet completely explored):

- Forensic and economic (legal investigations, good quality, illegal imitation).

Diagnosis and material qualification

Diagnosis and material qualification is obtained as an indirect information (or measure) from

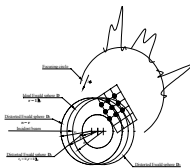
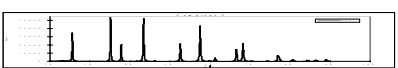
- identification of the lattice features from the **position of the diffraction lines**
- determination of the **peak intensity (or integrated intensity)**
- Evaluation of the **profile shape** of the diffraction line (Gaussian, Lorentian or overlaps of those)

Large use of laboratory Diffractometers (Destructive Testing or almost DT)

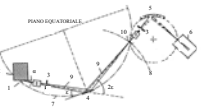

Rare use of Diffractometers for in place analysis due to the folloing main limiting causes

- Dimensions of the traditional Diffraction equipment
- Quality of the analysis
- Operators safety

Synthesis of a XRD Measurement

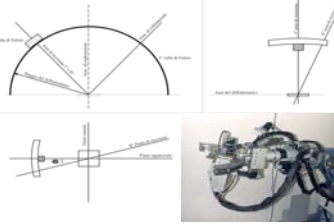



Traditional XRD

1. Fonte di luce
2. Filtro di onda
3. Condizionatore di temperatura
4. Campione
5. Rivelatore a Contatore scintillografico
6. Contatore di impulsi
7. Amplificatore
8. Registratore

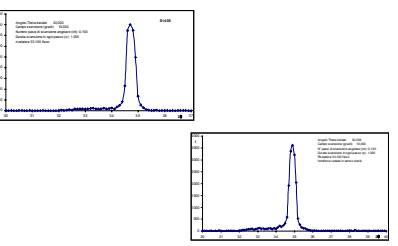
Portable (mobile) XR-Diffractometer



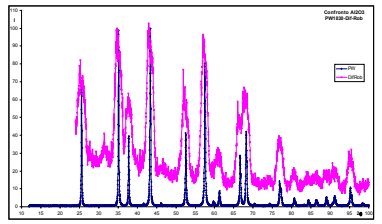
Filmato Dif-Rob

Current State of the Art

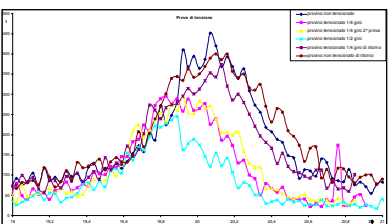
Results from a Silicon (Si) specimen with planes in the direction [4 0 0].



Comparison between data collection (normalized at 100) from specimen of polycrystalline Alumina (Al₂O₃) by a traditional laboratory diffractometer (blue line) and the movable diffractometer "Dif-Rob" (pink line).

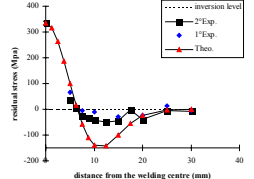


Test of Mechanical Load (applied Stress) on a Aluminium specimen



Theoretical and experimental values

Residual Stress (Mpa) vs. distance from welding .



Filmato Dif-Rob