STUDIO D'INGEGNERIA ROGANTE
ROGANTE ENGINEERING

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Engineering knowledge and applied research managing support in the neutron field for industrial companies and scientific institutions

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Abstract - 1

Ever-increasing performance and feature requirements for industrial components

➔ need for enhanced quality of the manufacturing procedures

➔ need to supply industrial companies with additional knowledge on innovative methods of analyses, new management criteria and appropriate interactions with large-scale facilities

Neutron methods = progressively significant probe for materials across many disciplines: increasingly helpful in studying materials and parts of industrial interest, allowing to monitor key parameters helpful to assess the origin of failure, enhancing quality of manufacturing processes and further supporting life assessment

ESS will only cover ≈20% of all the needs:
➔ design studies for new neutron sources and new improvements in the optimisation of neutron instruments, techniques and data treatments
➔ need to focus on completing neutron instrumentations with dedicated devices suitable for industrial applications

Abstract - 2

- Rogante Engineering Office (REO) has pioneered **Industrial Applications of Neutron Techniques** in crucial industrial sectors: some representative applications are described
- A model of interactions between industrial companies and large-scale facilities is reported with reference to the coordination of the WG13 "Industry and Industrial Applications" of ESS-Italia carried out by REO and related to the preliminary phase of the ESS project
- The cooperation of REO with scientific institutions for the development of new neutron instrumentation is reported

From the official web site: http://www.roganteengineering.it/AITN2008_web.pdf

REO: main activities in the nuclear field

- Reference point for Industrial Applications of Neutron Techniques
- Consulting for Decommissioning of NPP and Nuclear Facilities
- Project and consulting for aeration and filtration systems for NPP and Nuclear Facilities
- Coordinator of Italian Industry in the preparatory phase of the ESS project
- Consultant & Supplier of Industries and Bodies at international level

Stages from early research to market uptake - 1

Ever-increasing performance and feature requirements for industrial components:

➔ enhanced quality of the manufacturing procedures and development of advanced materials, to improve industrial products both in efficiency, quality and price

➔ need of special ability to manipulate material's properties at the atomic level

➔ neutron science is a key to these efforts

Key Issue:

To supply industrial Companies with additional **engineering knowledge and applied research managing support**

A priority target for the Regulators:

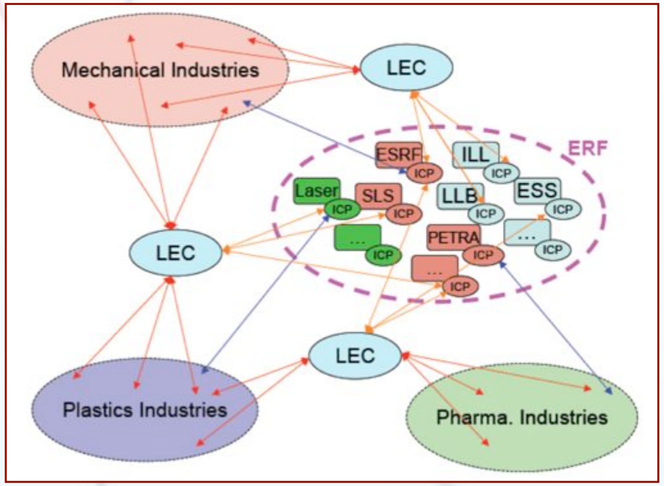
To support industrial Companies by finalising contracts with appropriate regional "Liaison Engineering Companies (LECs)", able to couple an industry in need of service to the most appropriate applied research strategy. In this case, the role of a LEC is essential to develop the industrial need into a possible industrial application.

Stages from early research to market uptake - 2

To implement this scheme, the Regulator (e.g., EU) could finalise contracts with appropriate LECs for scouting and networking activities

The advantage would consist in a large and professional coverage of diverse fields of Industry and geographical regions enhancing the number of potential cooperations.

The outreach would be drastically developed



Schematic representation of the interactions between Industry and Research institutions (Ris):

- blue arrows indicate existing and future direct contacts between individual industry and Ris
- red arrows indicate interaction channels between industries and LECs
- orange arrows indicate measurement campaigns managed by LECs

REO provides to the Italian industrial Companies a support in studying and enhancing their materials and components, improving the successive phases of production

REO has long experience in the Industrial field and in Advanced Research Methods (ARMs) of use to the specific Industry fields. REO has also established personal interactions with the key people of the Italian industries with potential applications of neutrons.

Example 1: coordinating Industry and Industrial Applications for Neutron Facilities

REO, as "Liaison Engineering Company", has been chosen to coordinate in Italy the **WG "Industry and Industrial Applications"** of ESS-Italia (a Company founded by the CNR, the INFN and ELETTRA) during the preparatory phase of the ESS Project.

ESS - ITALIA http://www.ess-italia.it/WG13.aspx

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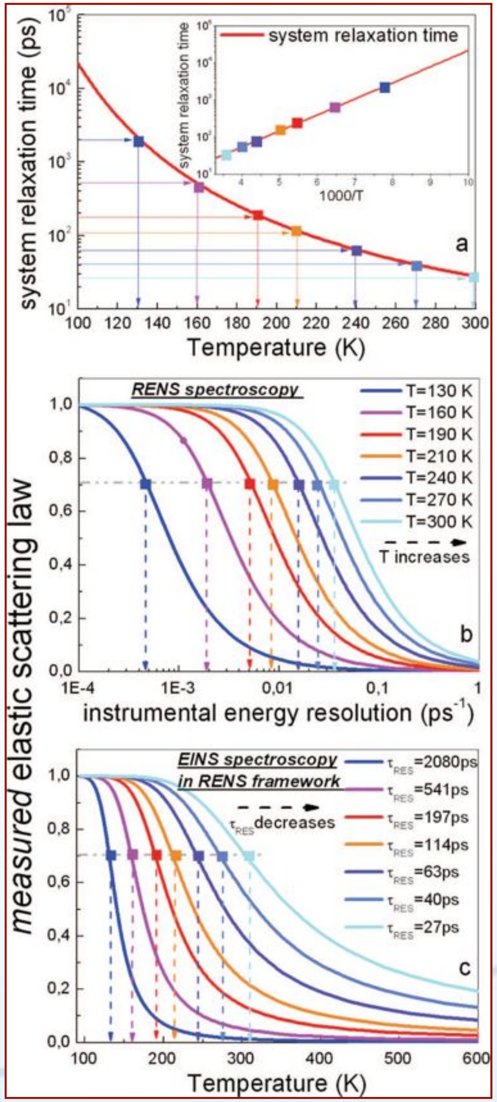
Example 2: Managing industrial providers and interactions for the development and construction of neutron instrumentation

REO, has been chosen by the University of Messina, Italy, to **manage industrial providers and interactions** for the development and construction of the Resolution Elastic Neutron Scattering (RENS) spectrometer, operating following the repetition rate multiplication mode

Italian participants:
Univ. Messina, Univ. Roma Tor Vergata, CISFA, Rogante Engineering Office, CAEN Vireggio, TNX Trento



The RENS method is based on the collection of elastic neutron scattering intensity as a function of the instrumental energy resolution and it is able to extract information on the system Dynamical properties from an elastic signal.
S. Magazù et al., Rev. Sci. Instrum. 82, 105115 (2011)

Neutron measurements data are particularly useful to solve industrial problems: key features and benefits for Industry

- ➔ becoming increasingly helpful in the nano-scale characterisation of industrial materials and components, revealing significant properties and allowing to monitor key nano-scale parameters helpful to assess the origin of failure, enhancing the quality of manufacturing processes and supporting life assessment.
- ➔ **REO, in cooperation with the BNC**, has pioneered Industrial Applications of Neutron Techniques in various crucial industrial sectors (see the BNC web page "Industrial Topics" at **http://www.bnc.hu/?q=node/33**).

Mail materials investigable by Neutrons:

Metals
Nanomaterials
Polymers
Composite materials
Shape memory materials
Glasses
Ceramics
Semi-crystalline systems
Membranes
Biomedical and Pharmaceutical materials
Gradient materials
Fibres

Solution of technical problems

Neutron Techniques can help in the solution of technical problems - connected to the performances of materials and components.
For instance:

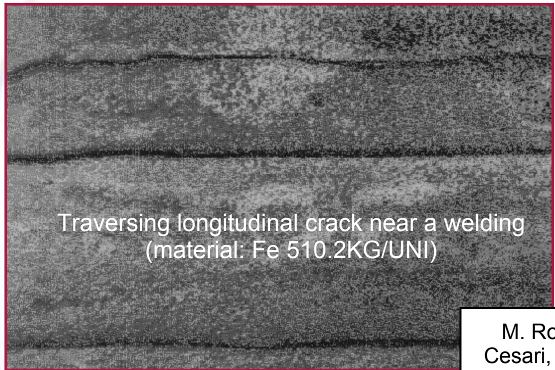
Fatigue and cracks…

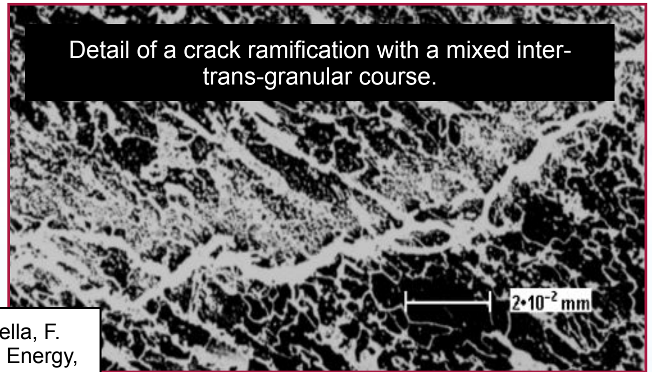
Fatigue = mechanism of failure → involves formation and growth of **cracks** under action of repeated stresses

→ cracks may propagate to such an extent that total fracture may occur

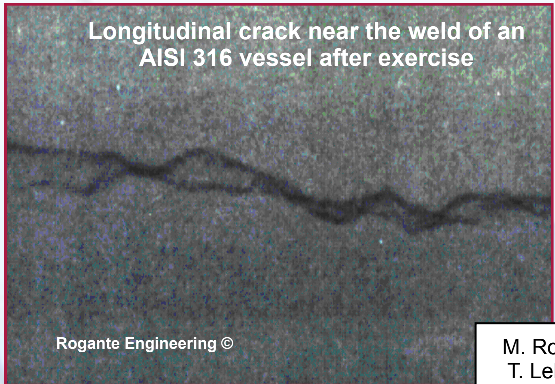
Cumulative damage assessment → fundamental to explore the wealth of research and its implications for design and manufacture

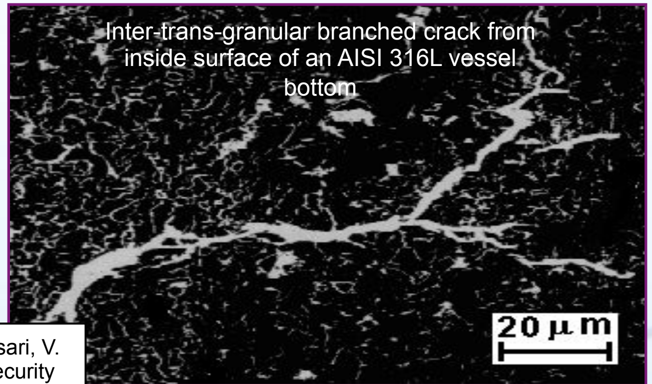
…material's failures due to cracks…





M. Rogante, P. Battistella, F. Cesari, Int. J. Hydrogen Energy, 31/5 (2006), pp. 597-601.





M. Rogante, F. G. Cesari, V. T. Lebedev, NATO Security through Science Series, Vol. XLI (2007), pp. 135-144.

...effects of forging processes...

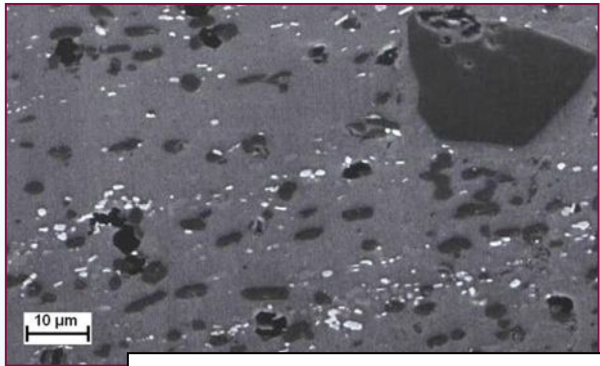
Mechanical forging ➔ high quality products by means of cold, warm or hot plastic deformations caused from collisions and pressures originated by forging machines

The main feature of the forged pieces consists in their **fibrous structure** corresponding to the orientation of the crystal grains and the fragmented phases.

The restoration mechanisms together with dynamic recovery and recrystallization can lead to **crystallographic texture and anisotropy of mechanical properties**, which consecutively could origin an orientation dependence of **residual stresses**.

➔ Stresses, strains, micro- and nano-structure play a key rule in forging

Alteration of mechanical properties in a Al alloy, due to forging: arrangement of precipitates reflecting the grain surfaces geometry



M. Rogante, L. Rosta, Strojarstvo 53/4 (2011), pp. 277-285

...Residual Stresses...

Residual Stresses (RS) → the stresses remaining in a material in absence of any external force.

They can be produced by the **manufacturing processes**, such as:

- extrusion
- welding
- forging
- plastic deformations
- thermal treatments

Stress Failure Modes

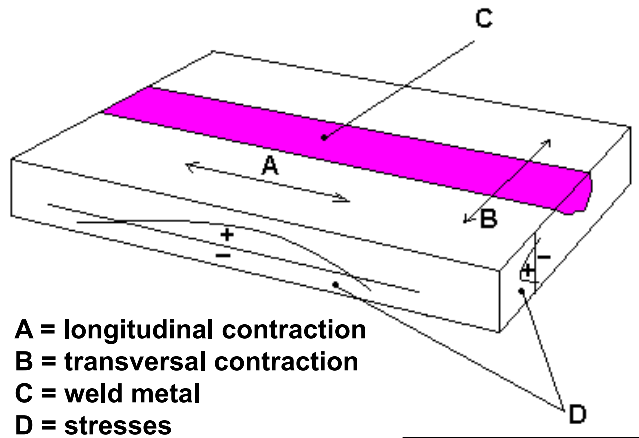
MECHANICAL RELATED GROUP	*MECHANICAL PROCESSING*, e.g.: - turning - milling - grinding
	PLASTIC DEFORMATION, e.g.: - rolling - moulding - drawing
	MECHANICAL TREATMENTS, e.g.: - shot peening - hammering
THERMAL/ METALLURGICAL RELATED GROUP	*WELDING PROCESS*
	THERMAL TREATMENTS, e.g.: - hardening - cementing - nitriding
	COATING PROCESS

Knowledge of the spatial and directional distribution of RS → **fundamental to determine their influence on the material and its performances**

...residual stresses due to welding...

Welding → inhomogeneous heating, microstructural transformations → complex and multi-dimensional **RS** distribution in the joint

The high temperature gradients produced during a welding process, generally, create some RS which can reach the yielding stress level

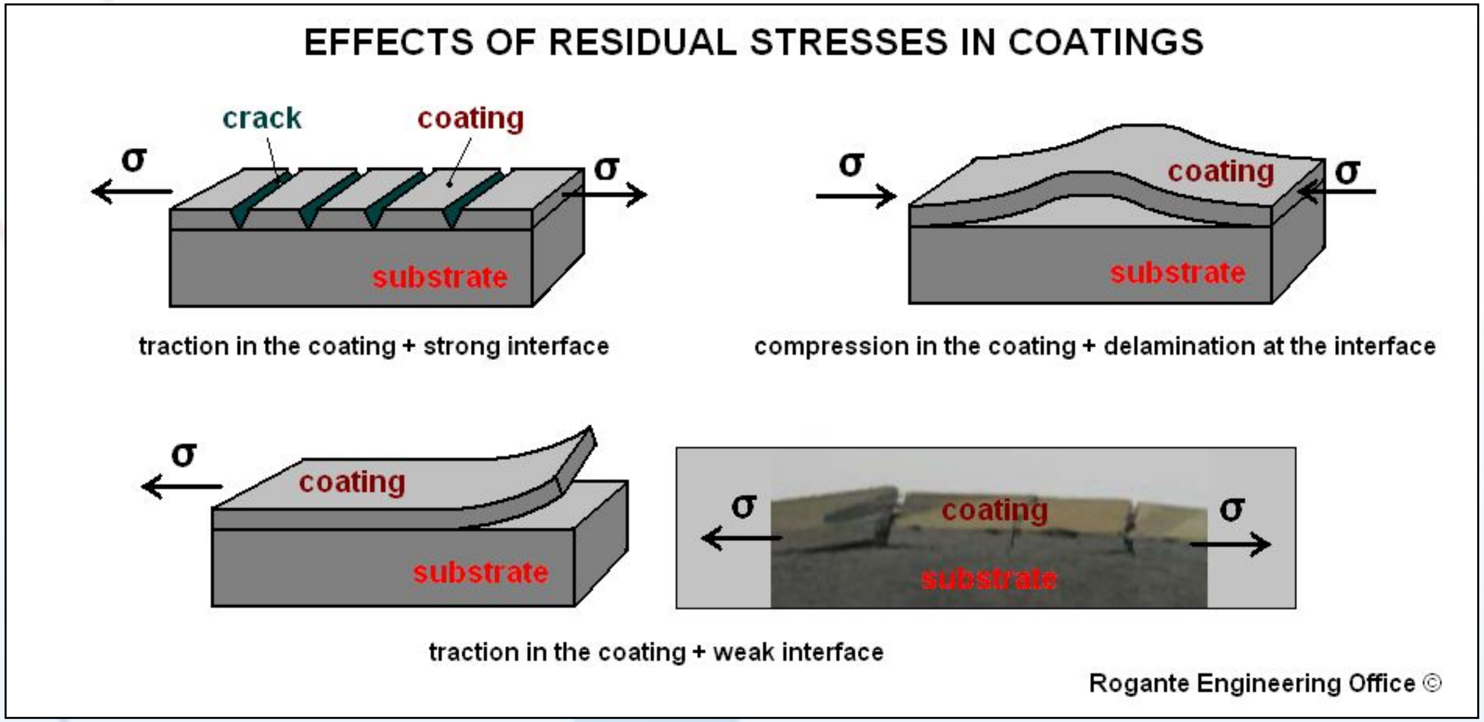


M. Rogante, V. T. Lebedev, S. Kralj, L. Rosta, Gy. Tőrők, MMMS, 2/4 (2006), pp. 419-433.

Contraction of molten weld metal throughout solidification is opposed by colder surrounding metal; therefore, some RS are induced

Knowledge of the spatial and directional distribution of RS → fundamental to determine their influence on the material and its behaviour

...Residual Stresses induced in the substrate by the coating process: they play a significant role in the coating behaviour...



...ageing mechanisms...

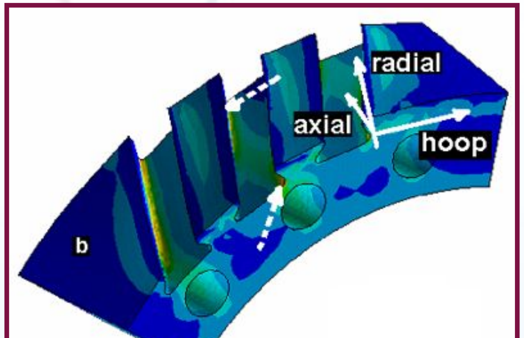
→ predisposed to reduce components and materials life:

- thermal and vibrational fatigue
- thermal ageing
- stress corrosion cracking (SCC)
- boric acid corrosion
- atmospheric corrosion

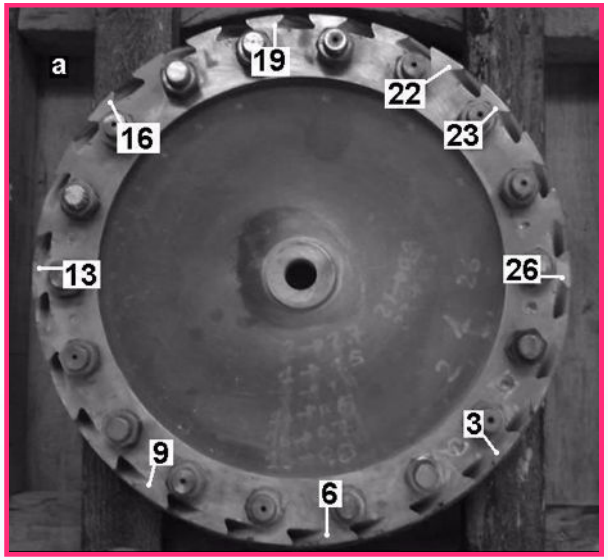
Ageing management of industrial components and materials ↔ in depth understanding of interactions between component design, materials, manufacture, operating environment and functioning transients

- The information supplied by classical fractography is not enough to investigate **long term exploited materials and their ageing**
- SANS allows characterising very small precipitates at early ageing time while they are not yet detected by other methods

Residual Stresses determination in a wheel of the axial compressor of a heavy-duty gas turbine



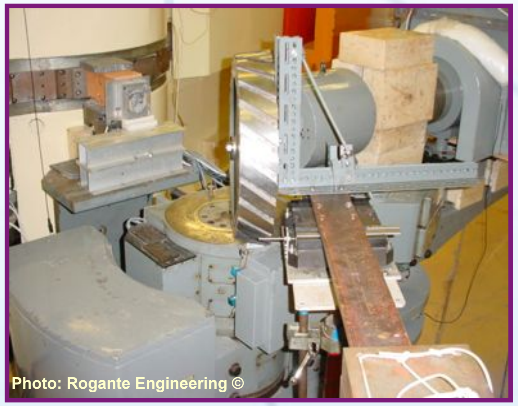
FEM Analysis



Wheel of the axial compressor of a heavy-duty gas turbine investigated by neutron diffraction for RS measurement in points marked by a previous FEM analysis

Material: NiCrMoV steel (with reference to the ASTM A 471-type 2 norms), usually adopted in the manufacturing of forged components for gas turbines.

Measurements: along radial, hoop and axial directions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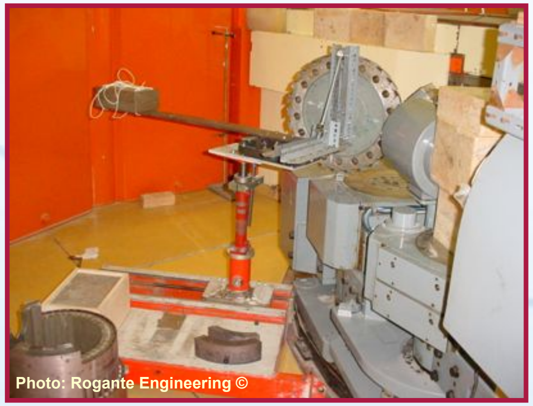
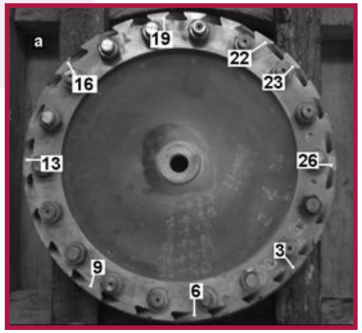
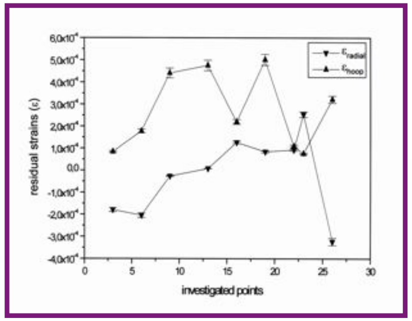


Photo: Rogante Engineering ©

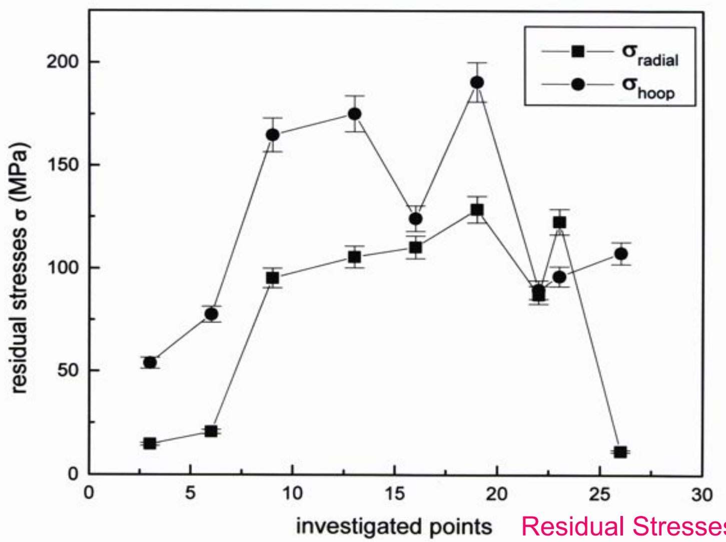
(Residual Stresses determination in a wheel of the axial compressor of a heavy-duty gas turbine)





Residual Strains

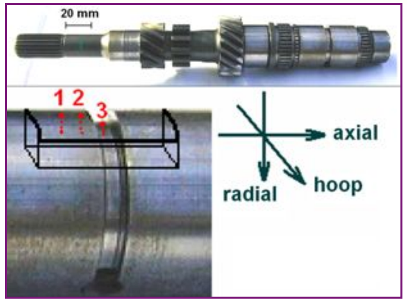
Investigated points	σ_{rad} (MPa)	σ_{tang} (MPa)
3	14,68	53,89
6	20,69	77,62
9	95,28	164,91
13	105,58	175,21
16	110,30	124,22
19	128,66	190,70
22	87,18	89,71
23	122,75	96,21
26	11,47	107,62



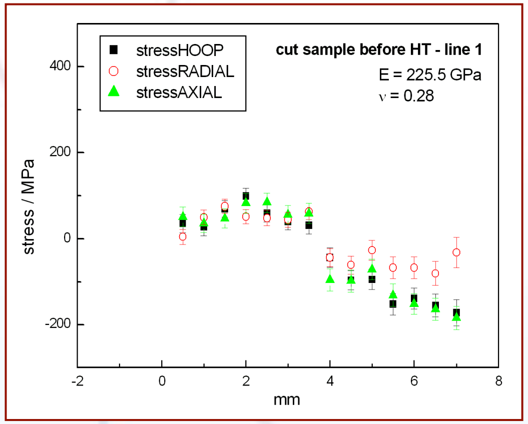
Residual Stresses

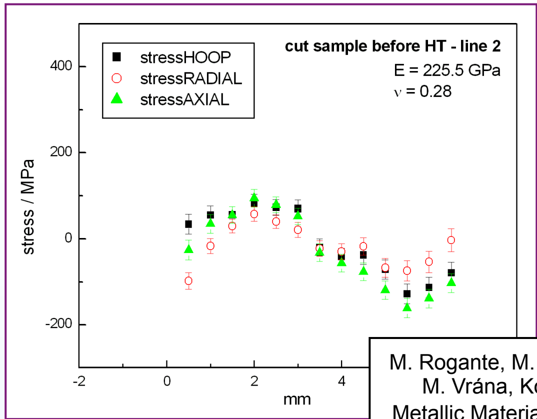
M. Rogante, G. Török, G. F. Ceschini, L. Tognarelli, I. Füzesy, L. Rosta, Physica B 350/1-3S (2004), pp. E479-E481

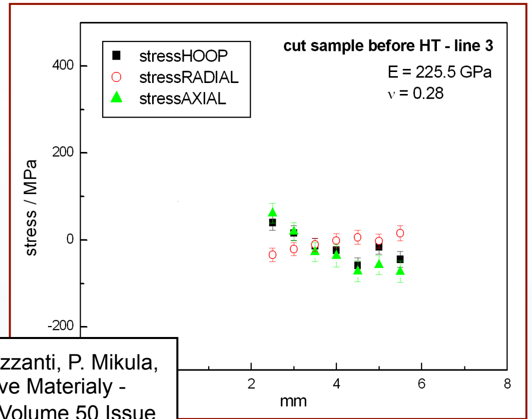
(RS determination in a car gear-shaft made of 20NiCrMo2 alloyed case hardening steel)



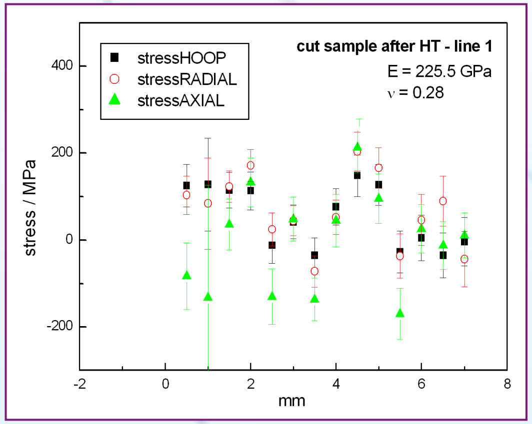
RS shown as differences between the RS status before and after the HT procedure

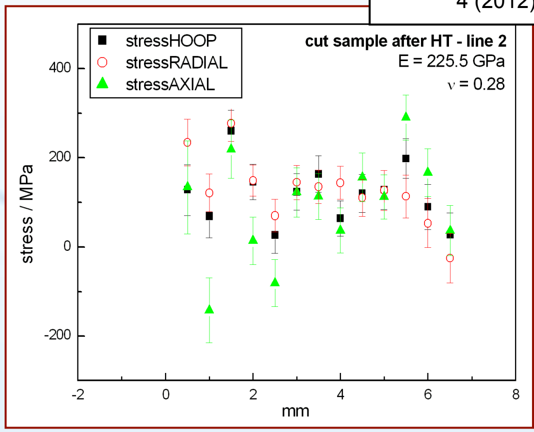


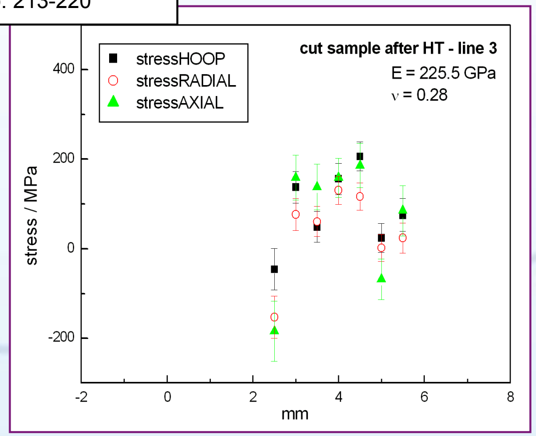




M. Rogante, M. Mazzanti, P. Mikula, M. Vrána, Kovove Materialy - Metallic Materials, Volume 50 Issue 4 (2012), pp. 213-220





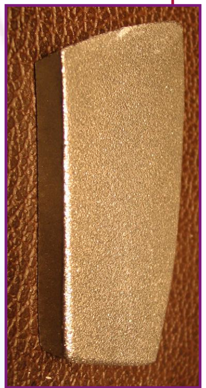


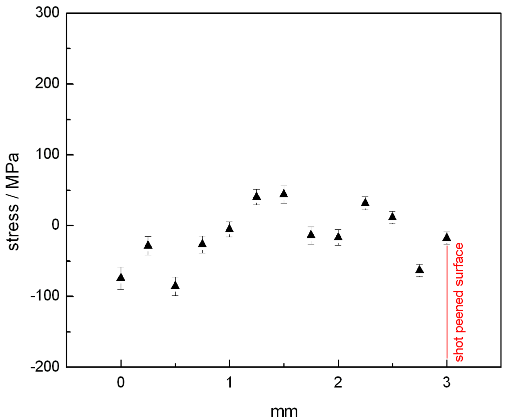
Near-surface residual stress determination in coatings

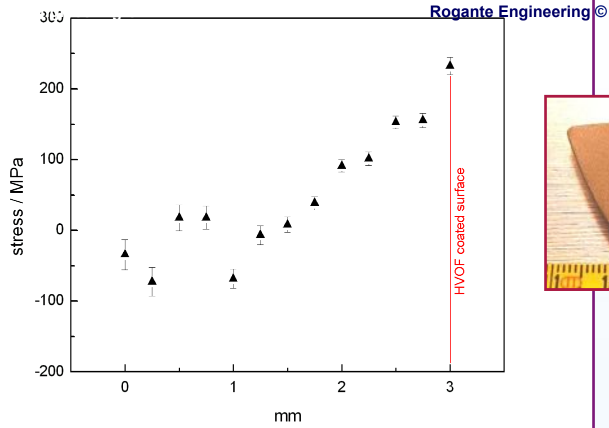
Two 13CrNi4 steel samples, one having a 0,3 mm thickness HVOF coated surface (88% WC + 12% Co matrix), the other a shoot peened surface
Through-the-thickness RS profiles determined
Complementarity between Neutron Diffraction and X-ray Diffraction

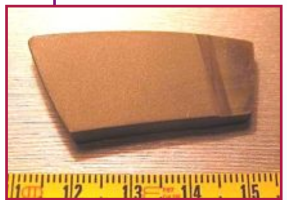
(Near-surface residual stress determination in coatings)

M. Rogante, P. Mikula, M. Vrana, Surf. Coati. Tech., 204 (2009), pp. 650-656.







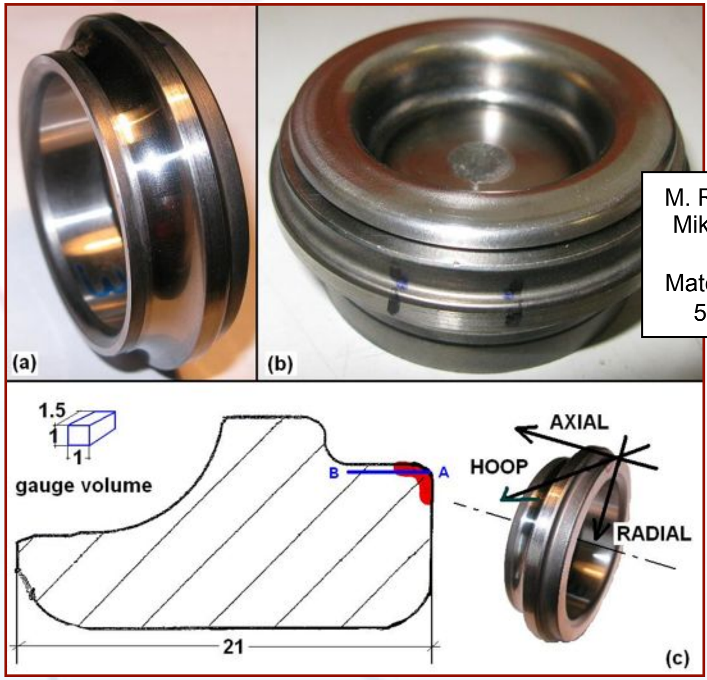


• **Uncoated sample**: RS in the range -80÷50MPa, i.e. no significant stresses through-the-thickness.

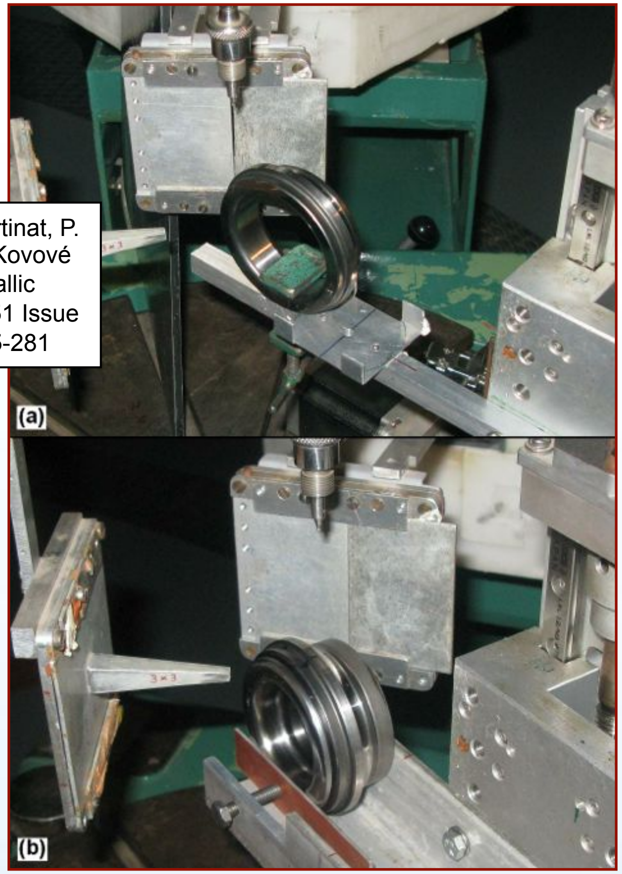
• **Coated sample**: RS increase gradually from a slight compressive status at the surface (0 mm) opposite to the HVOF coating, until the **greatest values of ~ 250 MPa at the HVOF coating** (3 mm).

• Complementary X-ray diffraction investigation on the coating surface ➔ compressive RS (σ = -550MPa±40), ➔ **potential risk in studying only by X-rays diffraction the RS in coated components:** a much different RS status could exist under the surface, most notably for non negligible stresses at a depth of 2 mm under the surface: additional ND investigation is recommended

Residual Stresses determination by Neutron Diffraction in a UNI 100Cr6 chrome steel ring



M. Rogante, G. Martinat, P. Mikula, M. Vrána, Kovové Materiály - Metallic Materials, Volume 51 Issue 5 (2013), pp. 275-281

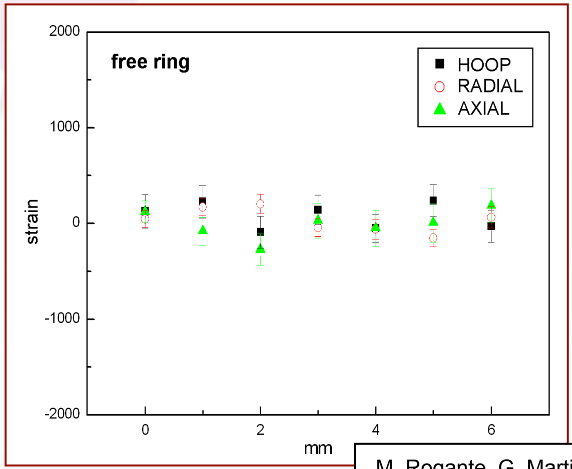


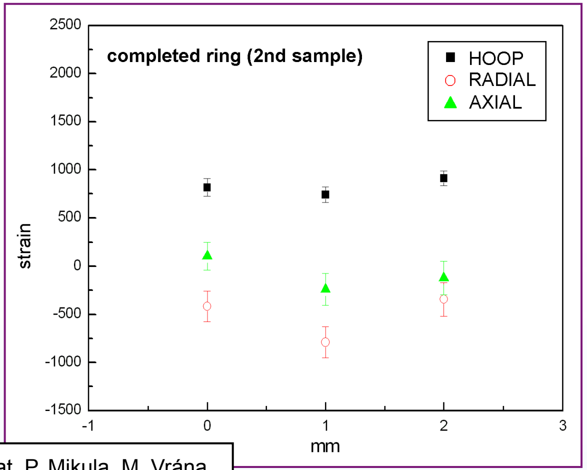
The free ring (a) and the same ring completed with the hub (b), during the ND measurements

UNI 100Cr6 chrome alloy steel: high hardness, high elastic limit, high fatigue strength, exceptional resistance to wear and deformation → specified by precision bearing producers where spherical and tolerance accuracy is essential

(Residual Stresses determination by Neutron Diffraction in a UNI 100Cr6 chrome steel ring)

free ring





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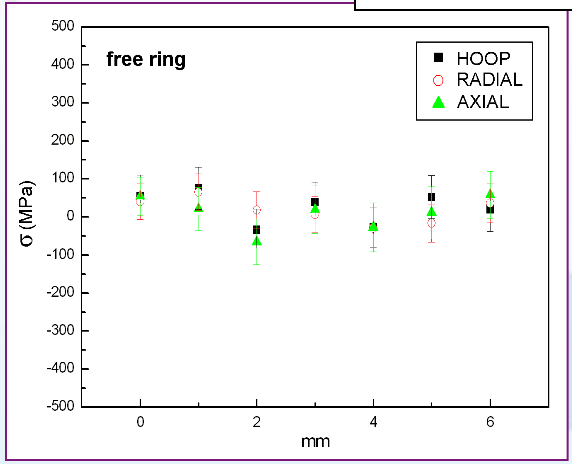
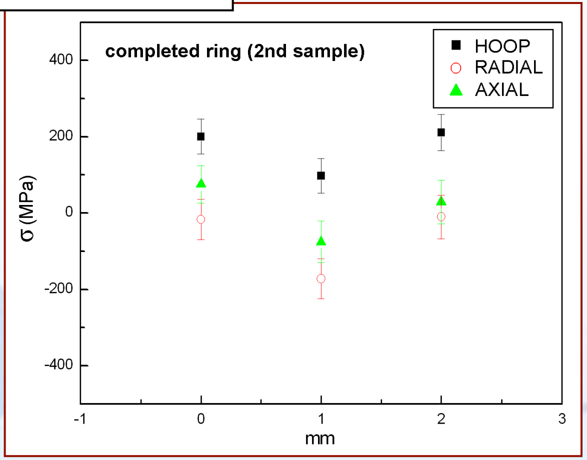
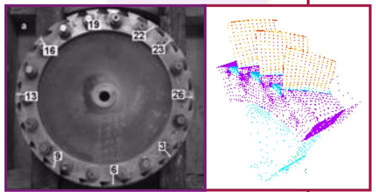
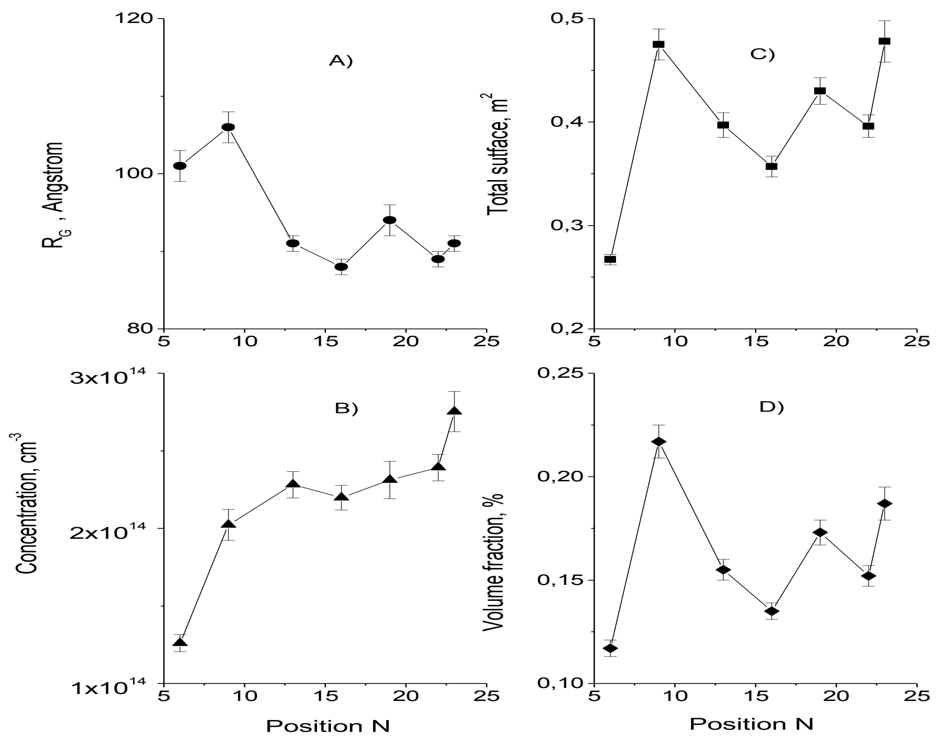




completed ring

Nano-scale investigation of a wheel of the axial compressor of a heavy-duty gas turbine





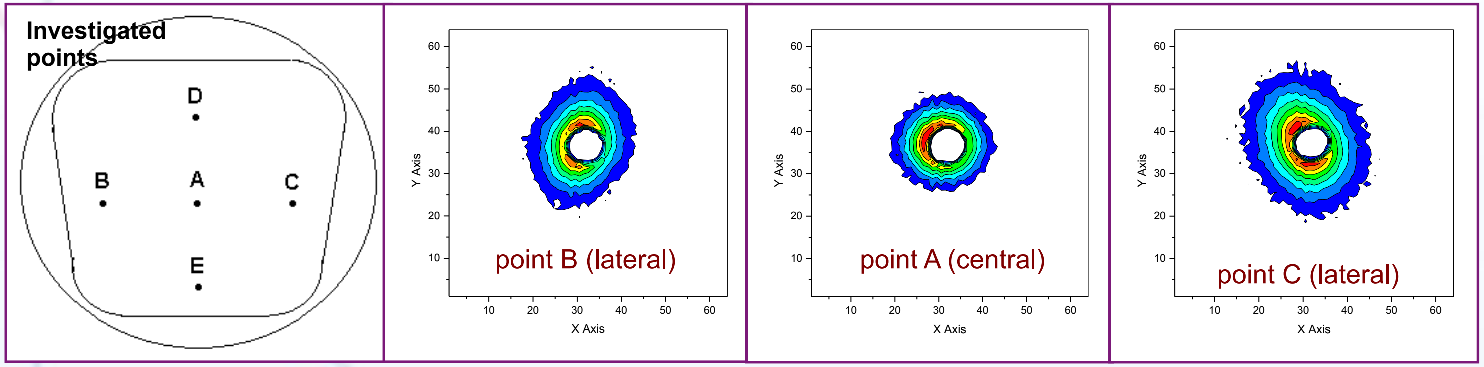
RESULTS OF SANS NANO-SCALE INVESTIGATION

The material has an isotropic nanostructure composed of : tiny domains (precipitates, **diameter** ~ 200-300 Å); **concentration** N≈(1-3)·10^{14} cm^{-3}; **volume fraction** φ ≈ 0.1-0.2 %; **total area of interface** St ≈ 0.2-0.4 m^2.

The SANS-data testify the material having a low concentration of **nano-defects**, as compared to steels after thermal treatment, where an intense formation is induced of precipitates which volume fraction exceeded ~ 1 %.

Nano-scale investigation of Al 4032 car engine pistons

- AlSi12CuNiMg alloy
- New and old pistons investigated
- Hot die forging performed in the range 370-510°C



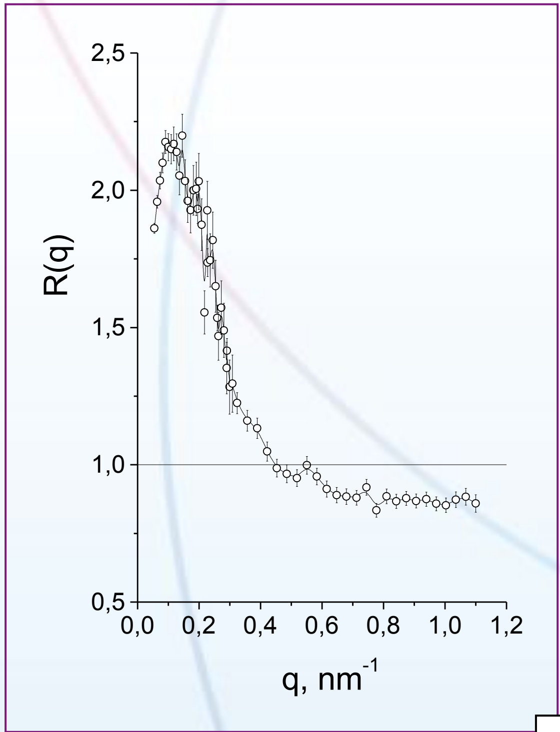
➔ Alteration of mechanical properties caused by the ageing treatment, compared with the precipitates size distribution changes

Problem of long-term materials exploitation under extreme thermal and mechanical stresses in car's engines: SANS can help studying the material state and predicting key phenomena ➔precursor of possible fracture of the engine elements

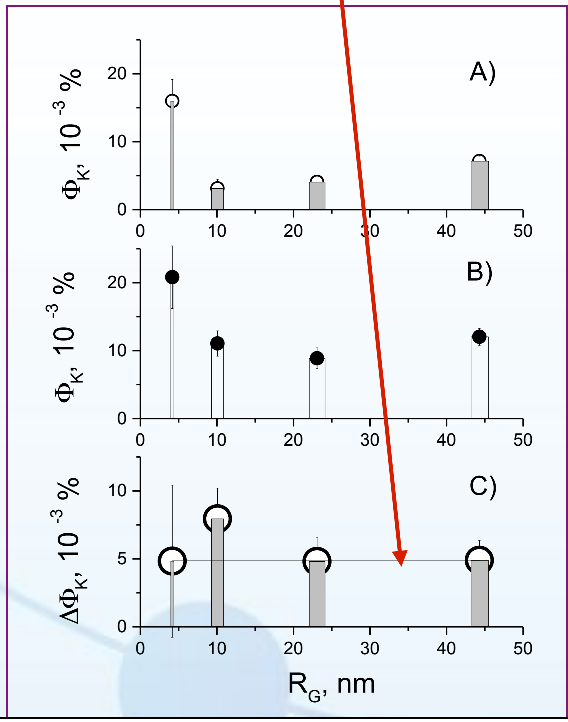
Isotropy in the central point and anisotropy in the lateral parts of the new piston crown ➔ presence of texture induced by manufacturing

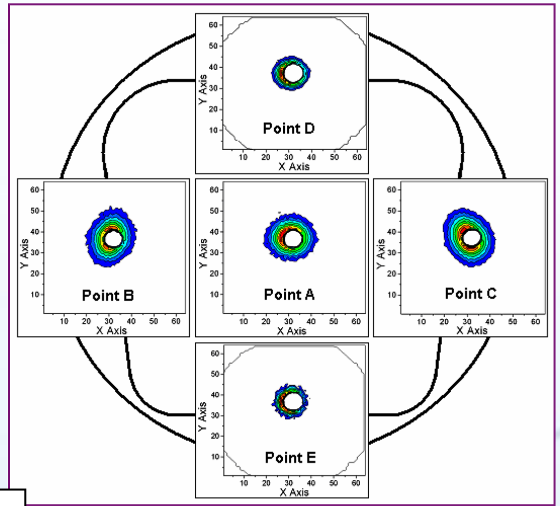
(Nano-scale investigation of Al 4032 car engine pistons)

Approximation $I_D = I_{OLD} - I_{NEW}$ representing the **nano-scale precipitates grown in the material**



Volume content of precipitates vs. their gyration radius: A) new piston; B) old piston; C) Difference $\Phi_{Kold} - \Phi_{Knew}$, line showing the average level for fractions 1, 3, 4





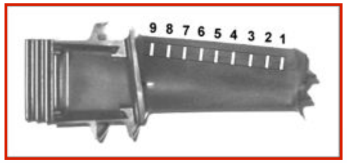
M. Rogante, V. T. Lebedev, F. Nicolaie, E. Rétfalvi, L. Rosta, Physica B, 358/1-4 (2005), pp. 224-231.

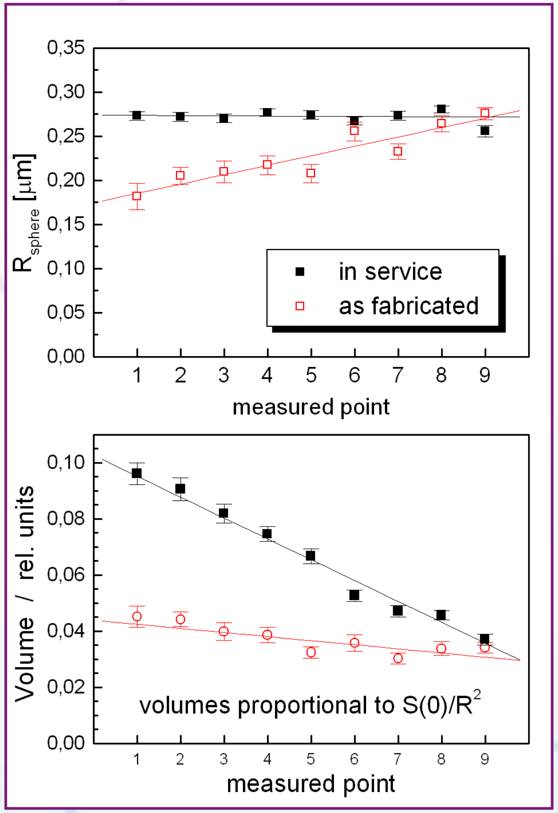
Nano-scale investigation of IN 738 turbine blades

- one blade after 25000 hours operation
- one blade not used

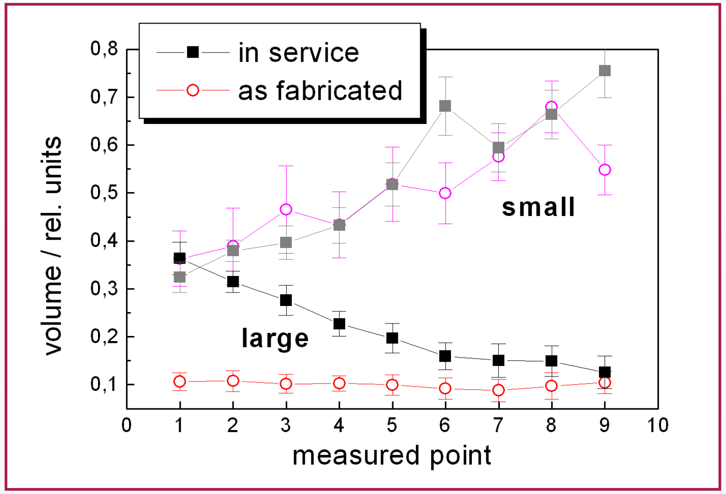
Each blade scanned (9 pos.)

Blade thickness: 3.5-5 mm





Effective sphere radii and volume fractions evaluated from Guinier approximation at small momentum transfers.



Integrated volume fractions (in relative units) of large and small precipitates.

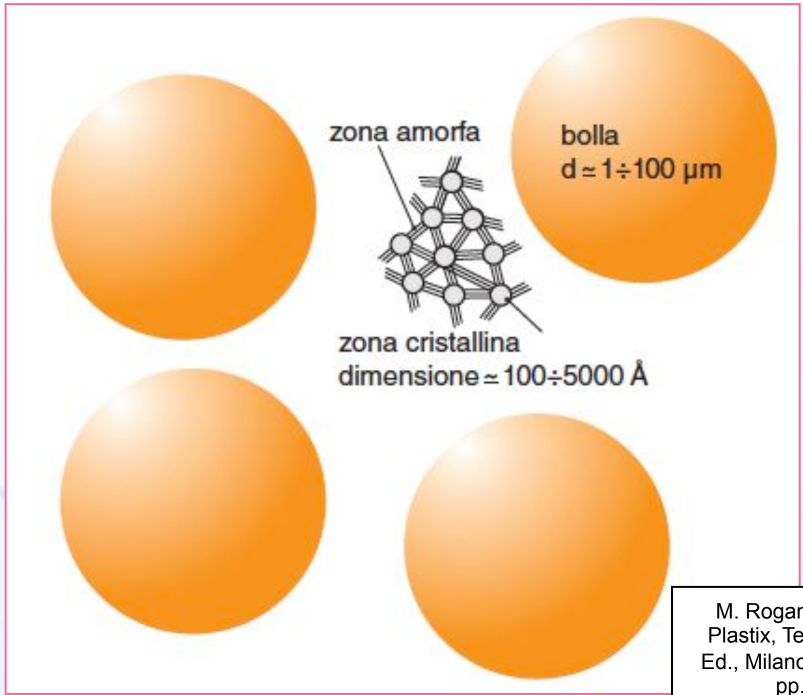
Growth of the particles at the tip of the blade during operation, probably due to high temperature levels, which induce the coarsening of turbine microstructure and growth of the precipitates.

Investigation of Polyurethanes

Polyurethanes are polymers containing the urethane linkage in their backbone chain

They are made by reacting Di-isocyanates with Di-alcohols.

Polyurethane mixture model. The dimensional scales are orientative



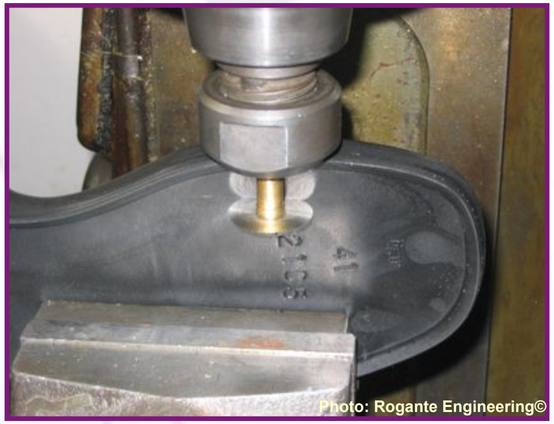
M. Rogante, F. Pisauri, Plastix, Tecniche Nuove, Ed., Milano, Vol. 3 (2015), pp. 46-51.

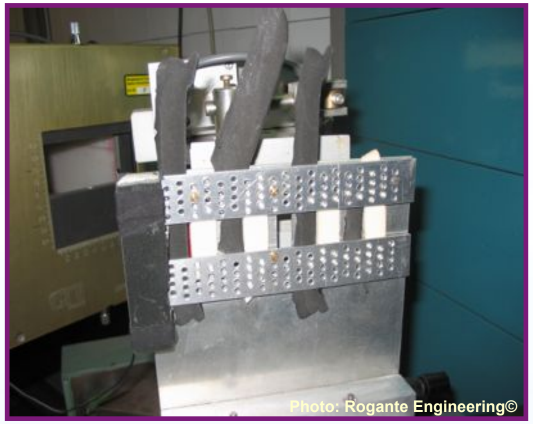
Amorphous zone →joining action between crystal zones

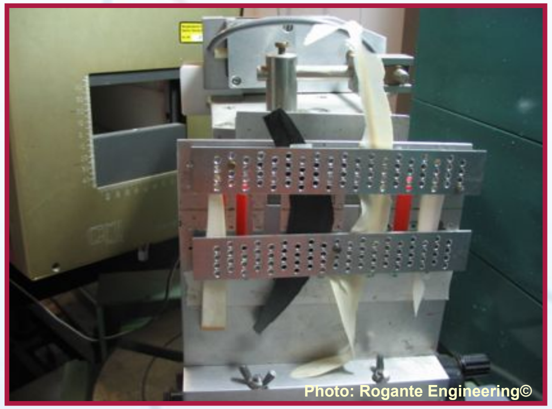
Bubbles - according to optical and neutron high-resolution data **-** and having a size ranging from 1 to 100 μm, can be closed or open, and they change depending on mixing procedure, catalysis, temperature and material type

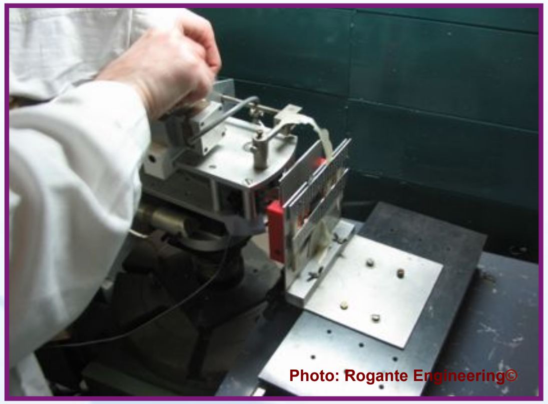
Crystal zone morphology → almost entirely ignored

(Investigation of Polyurethanes)





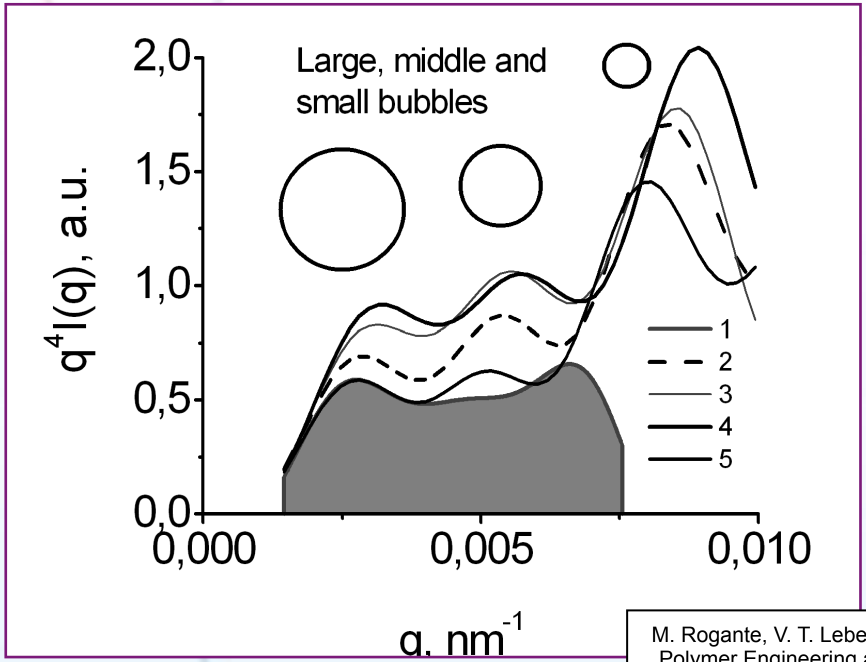




Samples preparation and SANS measurements carried out at the double-crystal diffractometer DN-2 at NPI Řež

(Investigation of Polyurethanes)

Scattering properties of the investigated polyurethanes samples - Fitting functions plots



M. Rogante, V. T. Lebedev, Polymer Engineering and Science, 47/8 (2007), pp.1213-1219.

Results:

presence of different fractions

total area of fraction vs. bubble's radius for samples of different technologies of polymer manufacturing

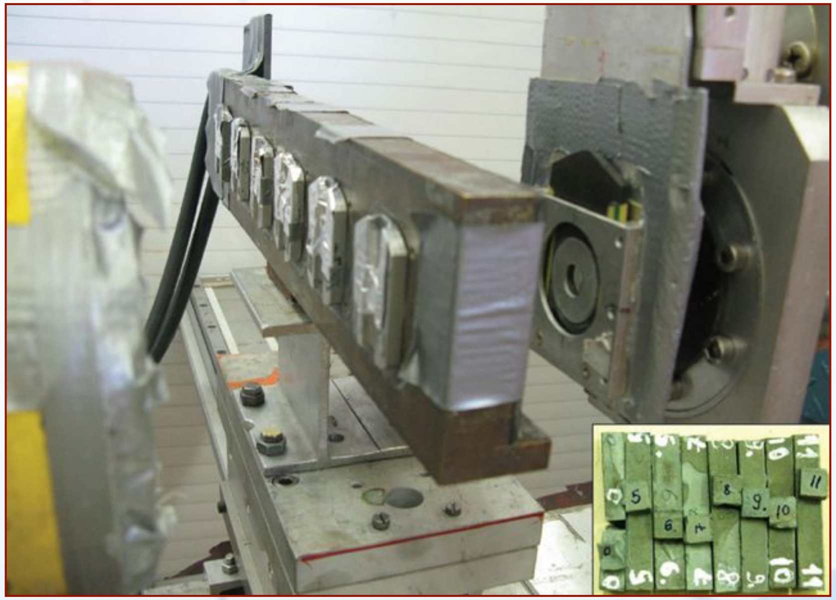
variation in chemical composition and technology of polymers → bubbles' radii change

The small fractions dominate giving a large contribution to the interface area

→ These results can really serve to control and predict the functional properties of polymers, which strongly depend on size and amount of defects and especially on their total area detected by SANS

Nanoscale investigation of Polymer Cement Concretes by SANS

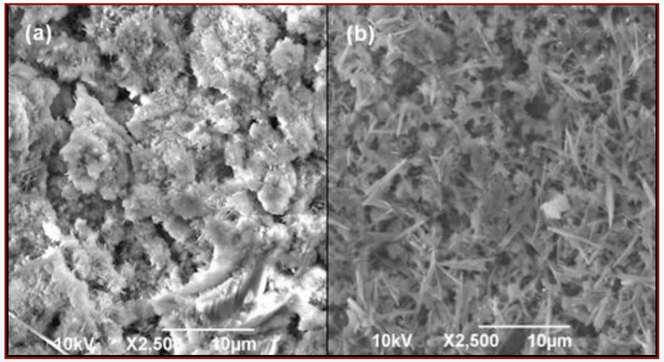
M. Rogante et al., Science and Engineering of Composite Materials (2015), ISSN (Online) 2191-0359, ISSN (Print) 0792-1233, DOI: 10.1515/secm-2015-0013.



sample	RDP (wt. %, type)	γAl_2O_3 (wt.)	mixing	sample thickness (mm)
S0	-	-	dry	3.28
S5	-	max	dry	3.65
S6	2.5 (RDP-22)	-	dry	3.69
S7	2.5 (RDP-23)	-	dry	3.66
S8	2.5 (RDP-22)	max	dry	3.45
S9	2.5 (RDP-22)	max	wet	3.64
S10	2.5 (RDP-23)	max	dry	3.72
S11	2.5 (RDP-23)	max	wet	2.88

max = maximum amount of nanostructured Al_2O_3

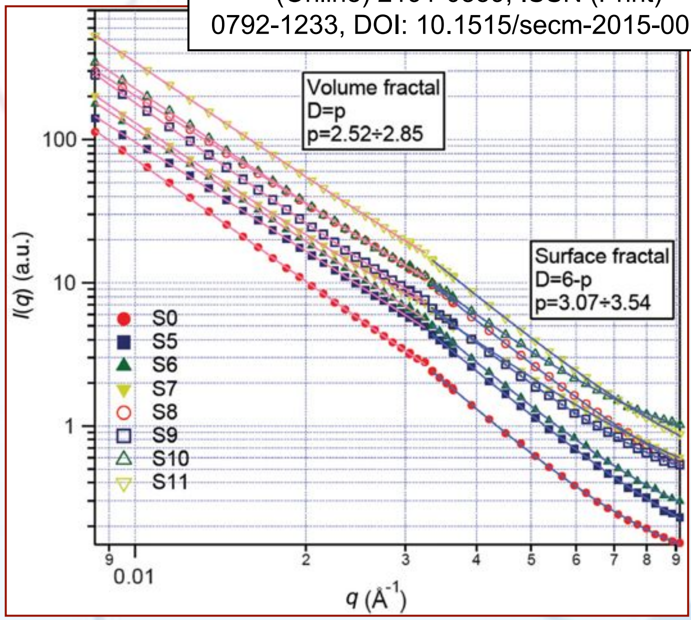
Composition and characteristics of the investigated samples made of Portland cement with added γAl_2O_3 and redispersible dry polymer



SEM picturesof PCCs without addition (a) and with maximum amount of nanostructured Al_2O_3+ 2,5% RDP-22 wet mixing (b)

(Nanoscale investigation of Polymer Cement Concretes by SANS)

M. Rogante et al., Science and Engineering of Composite Materials (2015), ISSN (Online) 2191-0359, ISSN (Print) 0792-1233, DOI: 10.1515/secm-2015-0013.



Averaged intensities from the SANS measurements represented versus the *q* scattering vector. The straight lines are the fitted model curves.

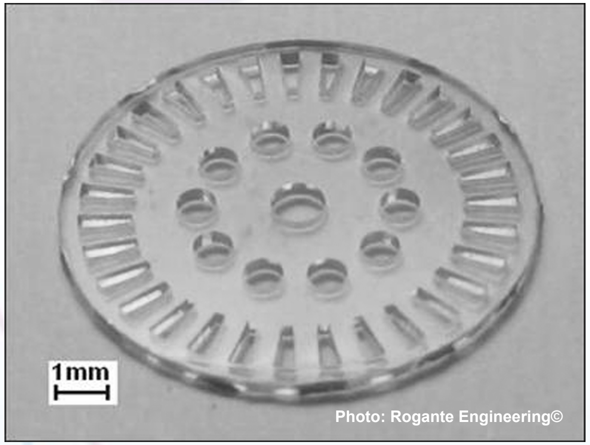
Some results

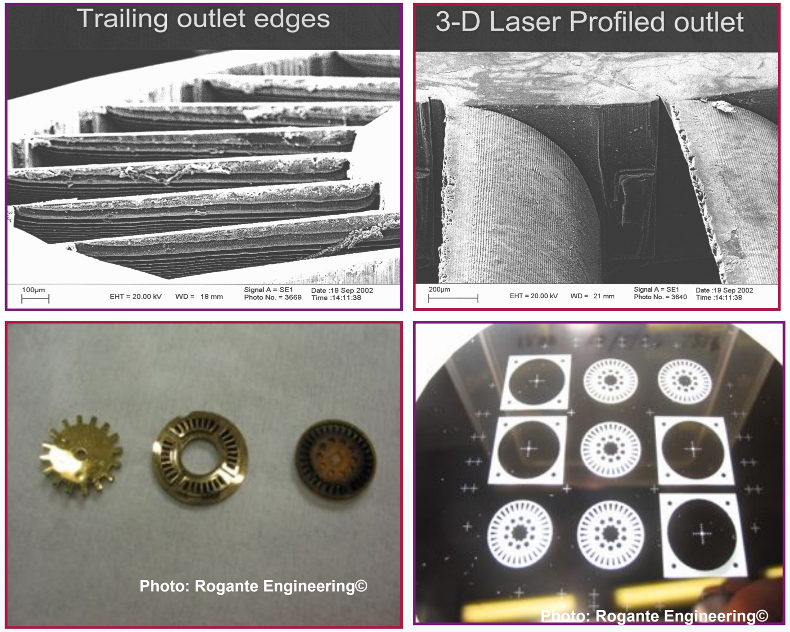
Each cement sample shows two different fractal regions.

When dry mixing is applied, the addition of mineral is necessary in order to achieve the optimum structural changes in the cement, namely to achieve the deposition of the polymer on the pores inner surface that increases the mechanical strength of the material.

The additives decrease the fractal dimension in the volume fractal region and increase the fractal dimension in the volume fractal region, meaning a higher structural stability of the system. The modification of cement creates preferably a smaller-sized structure of the cement stone.

Investigation of organic resin microturbines





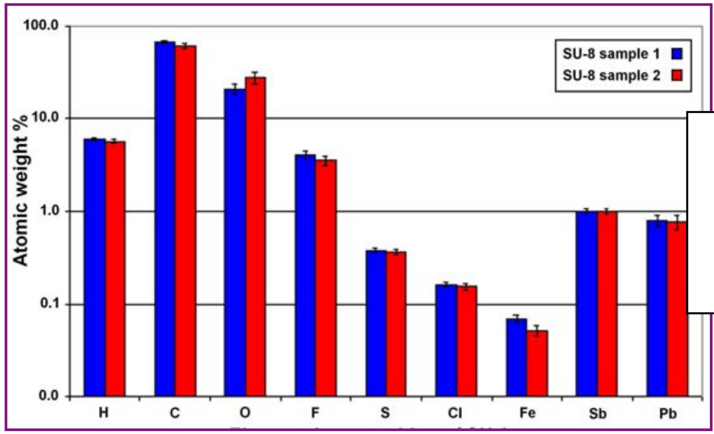
Innovative device to study gas and airflow using the low pressure-head features of axial-flow on the micro-scale: currently being developed from both a scientific and industrial point of view.

Constitutive material: **organic resin solution** that forms a negative photo resist including an epoxy resin organic solvent and gamma-Butyrolactone.

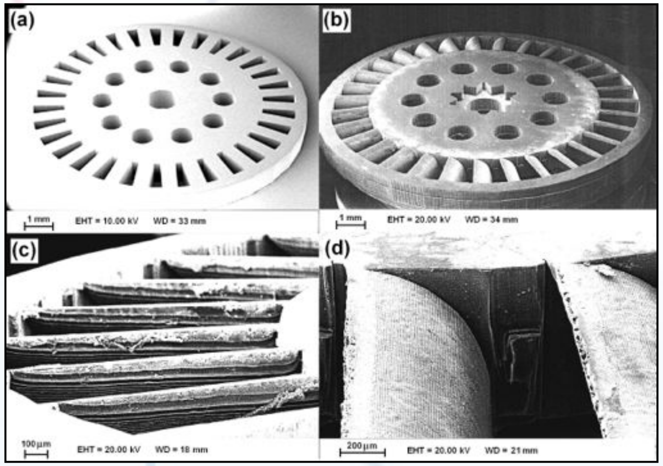
Resin constituents: Epoxy Resin (35-75%) and Gamma Butyrolactone (22-60%) mixed with Triarylsulfonia/ Hexafluroantimonate Salt and Propylene Carbonate (1-5%).

(Investigation of organic resin microturbines)

PGAA to check elemental composition stability under various material processing conditions
SANS to check the nano-sized objects inside the resin matrix

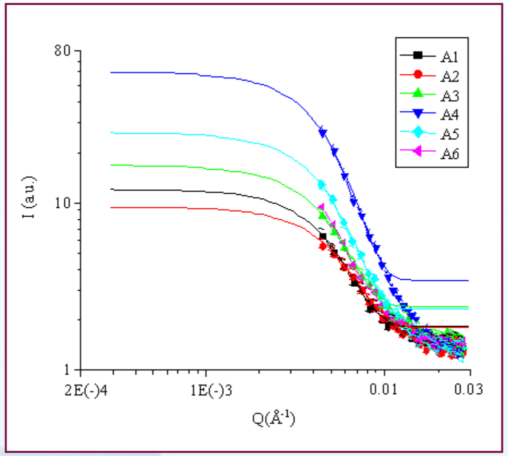


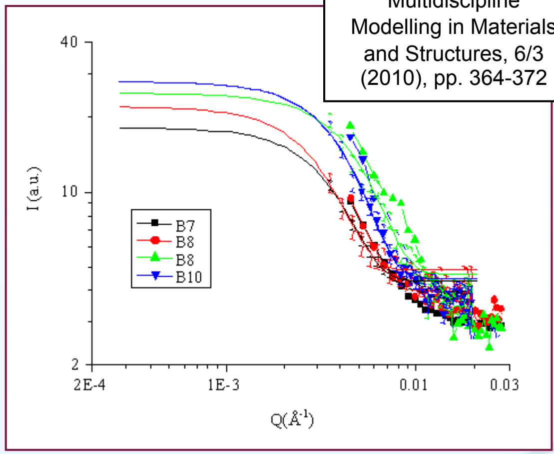
M.E. Heaton, M. Rogante, Zs. Kasztovszky, D. Denieffe, The Open Inorganic Chemistry Journal, 3 (2009), pp. 33-38.



(**a**) microturbine preform; (**b**) finished microturbine; (**c**) series of microturbine edges; (**d**) smooth surface detail of two turbine blades finished using laser machining.

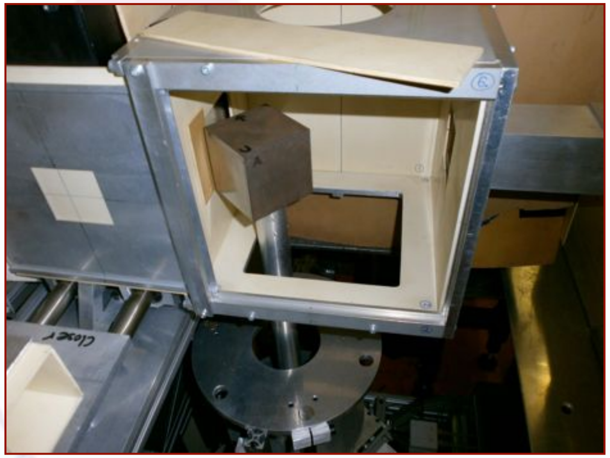
M.E. Heaton, M. Rogante, A. Len, D. Denieffe, Multidiscipline Modelling in Materials and Structures, 6/3 (2010), pp. 364-372



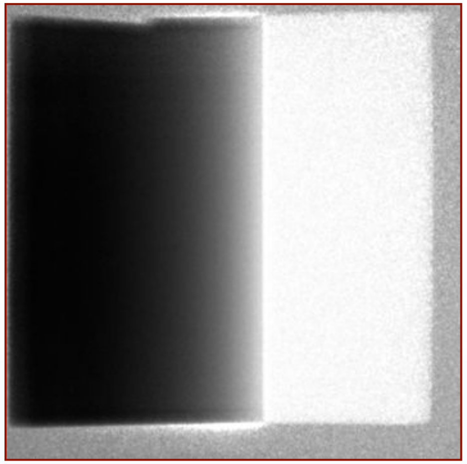


Samples A: defects size increase between 304 Å and 359 Å, showing that the laser ablation increased the size of the defects in comparison to the original rotor, while the aged rotors have larger cracks. The maximum sizes appear in the case of broken rotors.
Samples B: defects size increase with the growth of the PEB (post exposure bake) time - the maximum average defect size is 412 Å, that is 50 Å less then the breaking defect size of sample A5.

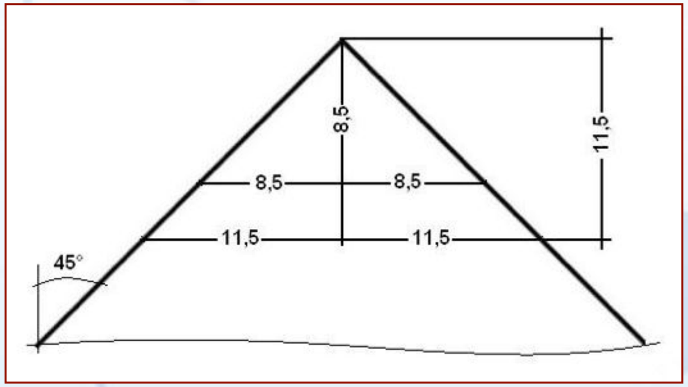
(Neutron imaging applications)



Neutron radiography (cold neutrons), ferritic steel cube at 45°.



Neutron radiography image (cold neutrons), steel cube at 45°, *texp*. = 1,8 sec.



Max thicknesses investigable that allow to obtain an acceptable contrast with reference to steel. In particular, at a thickness of 17 mm (8.5 + 8.5), about 1/5 of the neutron beam is detected, while at a thickness of 23 mm (11.5 + 11.5) about 1/10 of the neutron beam is detected

This experiment allowed to assess the power of penetration of the cold neutron beam in the considered material.

End of presentation

Köszönöm a figyelmet!

Thank you!

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