Rogante Engineering Office: Italian landmark for Industrial Applications of Neutron Techniques

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Abstract. The Rogante Engineering Office (REO), nuclear and mechanical engineering office and reference point for Industry, operating in the neutron field since over 30 years is as a landmark in Italy for Industrial Applications of Neutron Techniques (Applicazioni Industriali delle Tecniche Neutroniche®).

For these applications, the REO has developed special dedicated methodological approaches and procedures, having a multi-decennial experience: in understanding the broad range of problems faced by Industry and the complementarities between Research Infrastructure Methods (RMIs) and the other techniques adopted in each considered circumstance; in identifying the feasibility for each potential industrial application and the most appropriate methods and facilities.

Numerous Industrial Applications and pioneering neutron experiments have been carried out by the REO, adopting instrumentations of different research reactors, related to a wide spectrum of industrial fields - e.g., automotive, building, energy, footwear, medical and welding. Some of these works have been selected by the responsibles of the NMI3 Project - Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy - in the frame of the EU 6th Framework Programme, as the first works: in the NMI3 report "Engineering, Archaeology, Earth Sciences and Environment"; representing Engineering in the European Neutron Portal.

Industrial Applications highlights can be considered, e.g., the by now well known nano(micro)-structural analysis of Formula-1 engine pistons, the neutron analysis of heavy-duty gas turbine parts for the Oil & Gas field, the advanced characterization of industrial polymer materials for composition and bulk integrity.

The REO organized in Italy the 1st Italian Workshop for Industry "Industrial Applications of Neutron Techniques", which involved the participants in a "full-immersion" in the world of neutron techniques for the investigation of industrial materials and parts.

Based on many years of experience with Industry and RMIs, the REO, as Liaison Engineering Company, was selected and committed by the Italian Research Council to coordinate Italian Industry for the preparatory phase of the European Spallation Source (ESS), i.e. the Workgroup "Industry and Industrial Applications" in the frame of ESS-Italia. The Project Committee has been formed by the Italian Research Council, the Italian Nuclear Physics Institute and the Elettra Synchrotron (http://www.roganteengineering.it/public/ESS-ITALIA-WG13.pdf).

Prospects for the future are focused on enhancing this trans-facility inter-cooperation in the fields of science and technology, continuing to exploit neutrons in developing investigation projects on different industrial subjects: thereby creating new benefits for Industry and Research.

Key Words: Neutrons, Industrial Applications, nano-characterization, Residual stresses.

1. Introduction

Industry, nowadays, moves more and more to perfect materials and products and to optimize the control parameters of the relationships between structure, production and quality. The influence of manufacturing processes on structure and properties of the involved materials is therefore subjected to continuous study, to achieve the various set objectives including obtaining better performance and energy efficiency.

Specific parts of modern constructions (e.g., the automotive ones) must increasingly respond to stringent requirements from an engineering point of view. This also implies special analyses and difectoscopy activities, aimed at quantitatively and qualitatively identifying and outlining the surface and internal defects of the materials used. Neutron techniques, in this context, have repeatedly proved to be really useful, offering substantial advantage in comparison with the classical analysis and fractography, helping to solve key problems associated with the methodological limitations of these methods [1, 2].

Almost 30 years ago, the Rogante Engineering Office (REO) addressed its main activity to Applications of Neutron Techniques (ANT) for Industry and Cultural Heritage, adopting mainly Small angle neutron scattering (SANS), Neutron diffraction (ND), Prompt gamma activation analysis (PGAA), Neutron radiography (NR) and tomography (NT).

SANS allows characterizing materials at the nano(micro)-scale level, providing statistically precise information of 10÷1000 Å sized inhomogeneities averaged over a macroscopic volume. Neutrons' scattering on the sample is comparable to that of the photons, as described by the neutrons' wave optics. Since neutrons interact with the magnetic moments of the atoms, they also allow exploring magnetic inhomogeneities (e.g., different phases), precipitates or voids. The following parameters relative to the scattering objects (defects), in particular, can be monitored in materials submitted to irradiation or thermo-mechanical treatments such as creep, fatigue and ageing: size, shape, concentration, volume fraction, area of interface and chemical compound. Key information on chemical or magnetic inhomogeneities such as matrix heterogeneities (gas bubbles, pores and precipitates) can be achieved, considering also the possible local deviations in composition and technology, supplying information on ageing and degradation. The specimen can be studied or measured any number of times after further usage and/or treatment. The theoretical bases of SANS technique can be found in [1-6].

ND is an excellent and complete technique for non-destructive and non-invasive strain/stress measurement. It is able to provide data also including the effects of undesirable thermal variations, which are problematic to be studied only theoretically. Knowledge of directional and spatial residual stresses (RS) distribution is necessary to understand material's behaviour, being a decisive factor for safety, quality and service life assessment. RS concentration should be avoided to increase fatigue life and keep away from material's ageing related phenomena. In a ND measurement, the interplanar distance, the lattice strain and the stress values can be determined, also with reference to the main axes of the investigated object. Further information can be achieved linked with the number of dislocations and therefore with the plastic strain of the investigated material. Any alteration in the full width at half maximum (FWHM) from the reference distribution on an unstressed sample may be correlated to the increase of plastic deformation. The plastic zone, thus, may be assessed as the area where the FWHM is over that for the unstressed material. The results, besides the evaluation of the tensile or compressive condition, allow studying, e.g.: the efficiency of post-welding relaxation treatments; the effect of further heat treatment (HT); the RS relaxation fraction at the work temperatures and after a long time; the RS concentration related to notches and cracks. The theoretical bases of ND technique can be found in [1, 2, 7-9].

PGAA is based on the detection of characteristic prompt gamma photons originating in (n,γ) nuclear reactions. Every atomic nucleus, apart from 4He, may undergo a (n,γ) reaction with diverse probabilities and the energies of the emitted γ-photons are characteristic for every given isotope. The gamma peaks intensities are proportional to the amount of a given isotope. This phenomenon consents to use a quantitative elemental (isotopic) analysis method known as PGAA or prompt gamma neutron activation analysis (PGNAA). The prompt gamma spectra are recorded by using a high purity Germanium detector surrounded with Bismuth Germanate scintillators dedicated to perform the Compton-suppressed measurement mode. The signals coming from the detectors are treated adopting a multichannel analyser and the spectra are usually evaluated by using a Hypermet-PC program, which has a proven remarkable peak evaluation performance. The element identification is based on a prompt gamma library. PGAA consents a quantitative analysis (mass ratios or equally weight-percentage ratios of elements) and a comparison between different analysed samples, performed by using a principal component analysis (PCA). The PCA technique is able to reduce the dimensionality of large datasets, increasing interpretability and reducing information loss. In this case, it evidences similarities and dissimilarities. The detection limits of PGAA depend on composition of the investigated specimen. These limits can be improved by increasing the acquisition time. PGAA, due to the high penetrability of neutrons, can provide an average composition of the inside material, i.e. of the specimen in its whole or its fragments. The theoretical bases of the PGAA technique can be found in [1, 10, 11].

NR and NT allow penetrating a sample through a neutron beam, which is attenuated by the analysed material including some light materials such as hydrogen, and lithium. The attenuation happens according to the basic law of radiation attenuation. The detection is performed by an imaging device. The obtained information is related to the material and the structure inside the specimen. These techniques let visualise and assess material's distribution within macroscopic volumes of cement's specimens, as well as defining water's movement through the investigated samples. The obtained images of structures are also helpful to validate conventional measurements. The theoretical bases can be found in various references, e.g. in [1, 12-14].

Several works have been carried out by the REO, involving these techniques, solving key problems related to different industrial sectors such as Aerospace, Automotive, Bio-medical, Building, Coatings, Footwear, Mechanics in general, Offshore, Oil & Gas, Pipeline, Polymers and Welding. In Chapter 2, some of these works are described.

2. Examples of Industrial Applications of Neutron Techniques

An example of RS assessment by ND in pipes concerns a 2.25 Cr1Mo ferritic arc welded pipe before and after relaxation HT [15]. Figure 1 reports the obtained hoop RS.

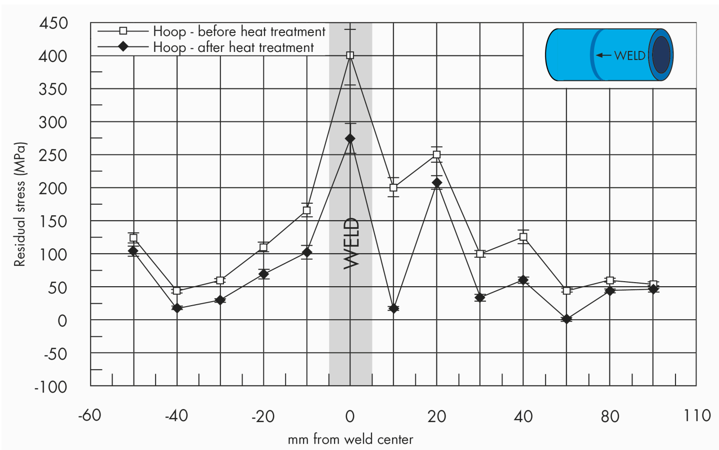

FIG. 1. Hoop residual stresses (5 mm depth) determined by neutron diffraction in a 2.25 Cr1Mo ferritic arc welded pipe before and after HT.

Neutron investigations of welded joints of neutron facilities can be performed concerning dismantled parts and new weldment made of the same constitutive materials. In weldment of austenitic steel pipes of nuclear power plants, delayed reheat cracking can occur in the heat affected zone, needing repair if major cracking arises. Such repair welds influence the RS distribution, which should be known to evaluate the effectiveness of the repair and to estimate the kinetics subsequent creep damage. ND, in this case, can supply crucial information and SANS allows verifying the fractal nature of structures and assessing the fractal dimensions [16]. This technique has provided crucial information on turbo-machinery parts and samples of constitutive materials, i.e. Inconel 738, Udimet 520 and Udimet 720 [17-19], characterizing precipitates dependently on temperature, hours of service and ageing time and evaluating parameters largely responsible for the functional properties of these parts. A new Inconel 738 gas reactor turbine blade and another after 25,000 hours of service were compared by high-resolution SANS at different positions along their edge, to assess the influence of high-temperature operation fatigue on microstructure. Size distributions and relative volume fractions of γ'-precipitates were evaluated from SANS spectra for each blade and position. As results, the precipitate volume fraction rises towards both blades tips. The old blade exhibits a substantial growth of precipitates average size and volume with respect to the as-cast blade, which can be explained by expected stronger precipitate coarsening under higher stresses. The precipitate size varies along the blade in the as-cast state. Figure 2 shows a blade during the SANS investigation at the adopted double-crystal diffractometer, as well as the obtained integrated volume fractions (in relative units) of large and small precipitates. Three populations of precipitates differing by characteristic size were revealed, exhibiting the larger differences along the scan in the used blade [20-22].

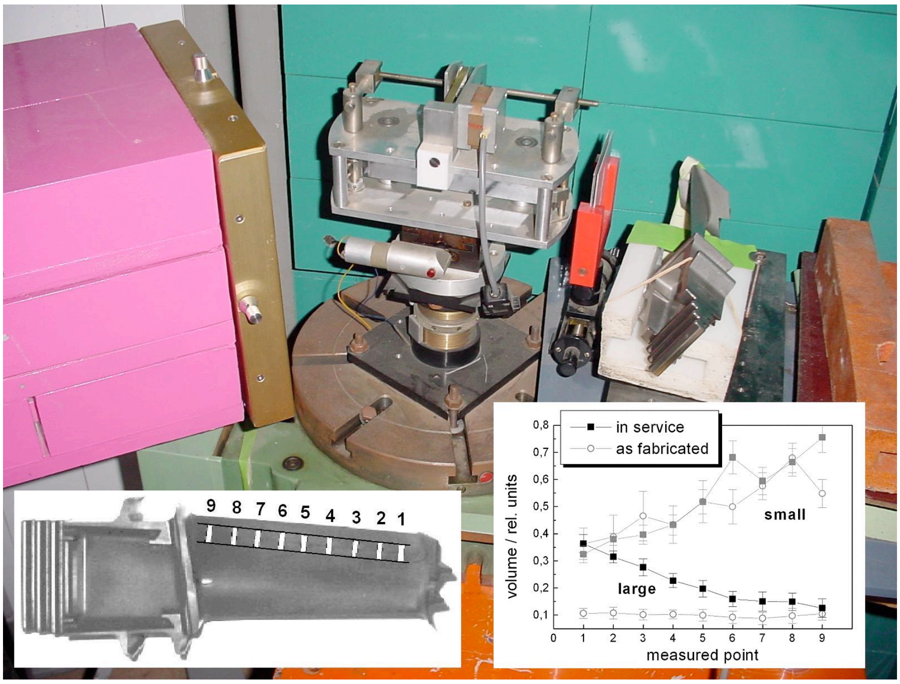

FIG. 2. Inconel 738 turbine blade during the SANS investigation and obtained integrated volume fractions of large and small precipitates.

A SANS study of precipitates' nano(micro)structural evolution was performed of the crowns of two Al 4032 car engine pistons (one new, another after operation). SANS curves were measured at five different points of both crowns, as possible critical locations: an isotropic scattering was observed at the central points of the new piston, while in the lateral parts the scattering became anisotropic, exhibiting a manufacture induced texture. Figure 3 shows the SANS patterns from the pistons crowns at the central position. In the precipitates ensemble, the growth of particles exhibited different rates, meaning the formation of different phases. The fractions of particles with radii R_G ~4; 20; 40 nm showed the same activity, while the particles with radii R_G ~10nm were growing twice faster. Such inclusions, having the volume content ~0.01 % can be related to strength and other properties by the material degradation under thermal and mechanical loading [23].

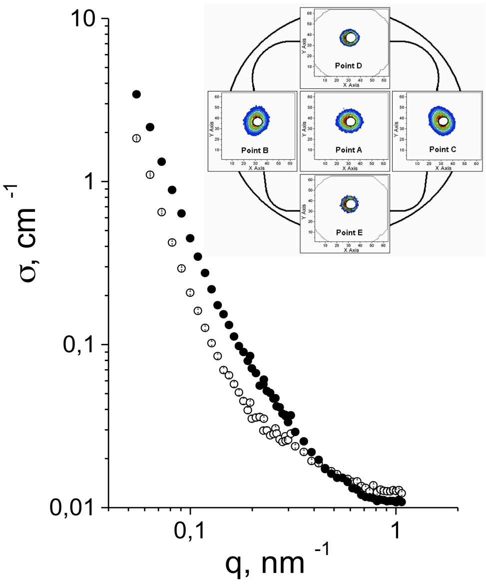

FIG. 3. SANS patterns from the piston crown at the central Point A: o = new piston; ● = old piston.

100Cr6 chrome steel submitted to martensitic hardening and tempering is usually adopted for bearings, rings and rollers. A 100Cr6 ring was investigated by ND before and after introducing a hub fixed by orbital rolling. The results supplied information on the real RS status, showing its enhancement due to the installation of the hub and the successive rolling operation [24]. NT has been adopted to investigate two 100Cr6 double-row ball bearings - one unused, the another damaged during use - applied for the belt tension lever of a car engine. The cuts of the tomograms showed several cracks in the inner ring of the damaged bearing and the missing lubricant near the damaged areas. The produced video (web link https://youtu.be/9of57wUy8X4) shows initially horizontal, and later vertical NT cuts at the damaged part, followed by a 3D rendering showing damaged bearing's internal cracks [25].

Polymer Cement Concretes (PCCs) are dense cements with improved functional properties such as resistance to crack formation and ageing, hardness and stability of mechanical modules. These PCCs have been considered also for the nuclear sector [26]. A SANS investigation was performed of PCC specimens made of Portland cement with different additions of γAl2O3 and redispersible dry polymer (RDP). Information on different nano-structural parameters was achieved, confirming that the modification of cement by these additions generates preferably a small-sized structure of stone, and in particular: the addition of small amounts of γAl2O3 or/and RDP makes more developed the interface between the nanoscale structural elements; these additions lead to a ~20 % decrease of the grains size; considering samples with added 2.5 % of RDP and with added 2.5 % of both RDP and Al2O3, the interface disappears, converting into a low-dimensional structure being mass fractal in geometry [27-29]. The *D* values shown in Figure 4 indicate in general a change in the interface quality (roughness) due to the introduction of γAl2O3 and RDPs.

A NiCrMoV steel wheel of a heavy-duty gas turbine axial compressor was investigated in nine locations of the teeth-section by ND for RS evaluation. Internal radial and hoop RS were determined, whose values resulted under the limit of 200 kPa: in particular, hoop RS resulted in the range of ~ 50-200 MPa, whereas radial ones were in the range of ~ 10-130 MPa [30]. Figure 5 shows the wheel during the ND investigation, as well as the RS values for radial and hoop directions.

A RS analysis by ND was performed of 20NiCrMo2 steel car gear-shafts (the whole shaft and a cut sample) in critical region (notch) sensitive to fatigue-stress and/or crack. The strain status before HT was taken as reference to determine RS after the HT and after the successive finishing process. Figure 6 shows the analysed parts and RS components determined in correspondence of line 2 (cut sample), as differences between the RS status after the finishing process and before the case-hardening treatment.

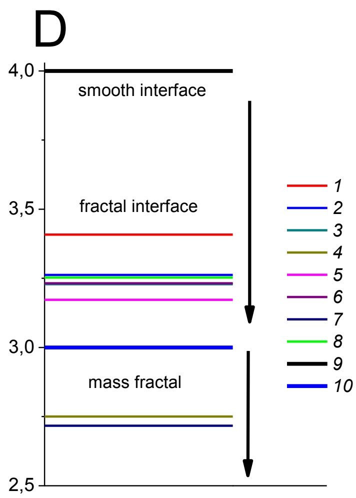

FIG. 4. Exponent D characterizing the fractal properties of interfaces between nanoscale structural elements, with reference to the 10 samples investigated by SANS.

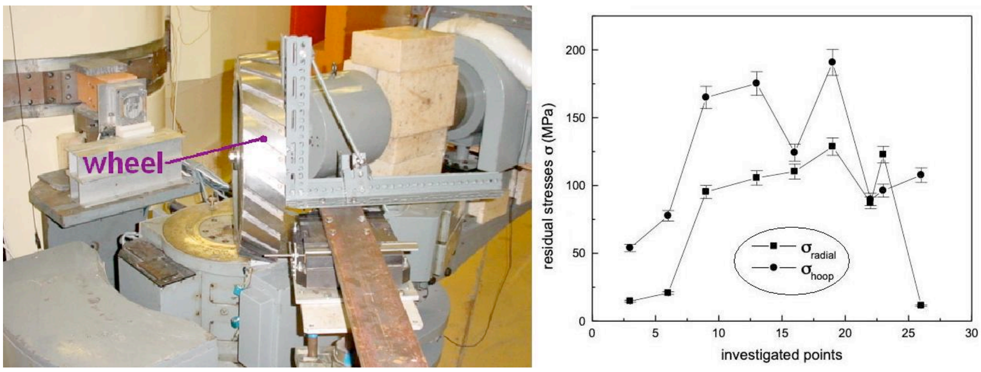

FIG. 5. NiCrMoV steel wheel of a heavy-duty gas turbine axial compressor during the ND investigation and obtained RS values for radial and hoop directions.

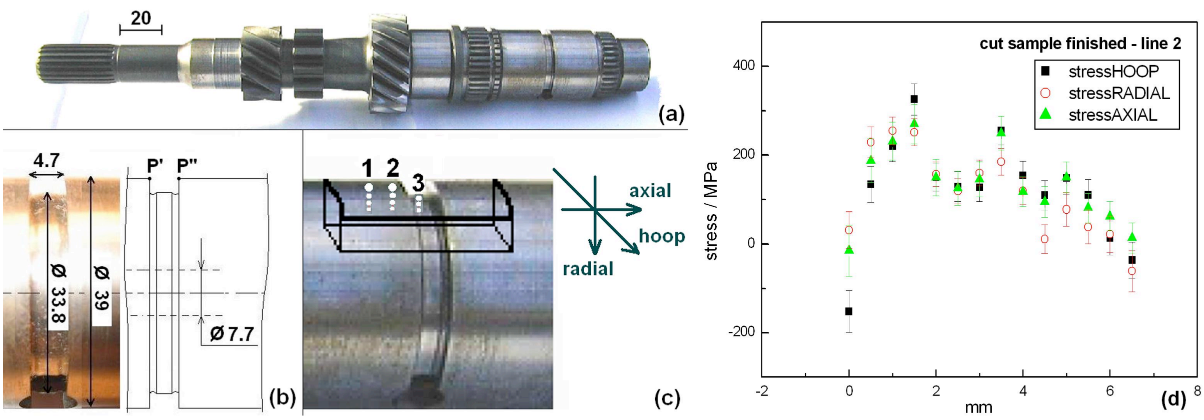

FIG. 6. 20NiCrMo2 steel car gear-shafts analysed by ND (a); points P' and P'' have zero axial and radial RS components (b); cut sample profile and investigated points (c); RS in line 2 (d).

Various other examples of Industrial Applications of Neutron Techniques carried out by the REO can be found, e.g., in [1, 2, 8, 9, 12, 21 and 22].

3. Conclusions

Neutron techniques are increasingly used in the study of industrial materials and parts, and their applicability is susceptible to new developments. The REO, having long since celebrated, e.g., 20 years of collaboration with the Budapest Neutron Centre and the Neutron Physics Laboratory of the CANAM infrastructure of Nuclear Physics Institute of the Czech Academy of Sciences and other nuclear related scientific institutions, will continue enhancing his inter-cooperations, exploiting neutrons in new forefront areas of science and technology.

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