The application of neutron examination to the pipeline industry

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THE USE OF NEUTRON techniques is becoming more and more widespread for nano-structural and atomic-scale studies of industrial materials and components, including those used offshore. Particularly in reference to permanent structures, such as pipelines, it is very important that the reliability of the welded joints involved is assured: the presence of even a micro-crack due to welding can produce yielding of the whole asset, with consequences that impact not only economically, but also from an ecological point of view.

In this paper, the basic theoretical aspects of neutron diffraction for residual stress (RS) measurement and small-angle neutron scattering (SANS) for microstructural characterization related to precipitates and inhomogeities in element distribution – among the major reasons for a joint's failure – are reported. Applications of both techniques are presented, connected with the pipeline sector, such as RS determination in pipe welding and SANS investigation of pipe materials. The information obtained can be used to improve the design, production, and maintenance phases, and contributes to the development of a nanoscopic safety criterion to forecast possible fracture processes, and provide assessments of quality assurance, safety enhancement, lifetime management, and cost effectiveness.

A VARIETY OF tools and methodologies is currently available for the assessment of the defects that can be introduced into a pipeline system during fabrication, installation, or operation – in particular, defective welds, pipe, and material, residual stresses (RS), and tensile corrosion – and their efficiency needs to be continuously evaluated in order to assess a pipeline's safety. Cracks are among the most dangerous kinds of defect in a pipeline, and the mechanism of their initiation and growth is still not completely understood. Stress-corrosion cracking (SCC) can arise in different forms from small isolated cracks to large crack areas, involving the risk of the pipeline asset yielding. Defects also occur while the pipe wall is under stress due to live pressure.

During the last two decades, the use of neutrons has become an increasingly significant way of probing into materials across a wide range of disciplines from mechanical engineering to biology, answering key industrial questions, and revealing significant properties of the materials. This success is mainly due to the peculiarity of neutrons, which allow the bulk of a sample to be probed, in contrast to x-ray techniques that are usually surface-sensitive. Industrial applications of neutron techniques in engineering and materials science mainly include:

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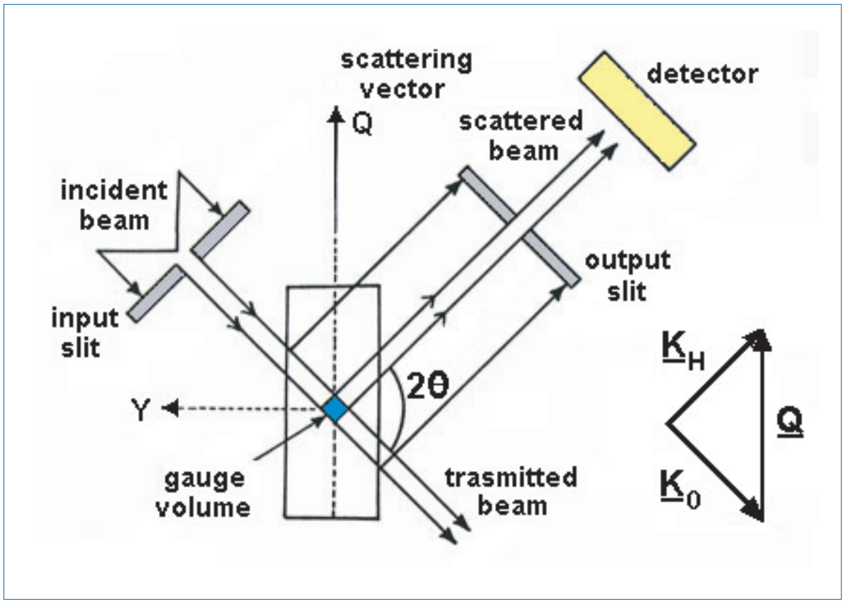


Fig.1. Schematic of strain measurement by neutron diffraction.

- RS determination and crystallographic texture measurement by neutron diffraction; microstructural characterization by small-angle neutron scattering (SANS);

- characterization by neutron activation analysis; analysis of the flow of fluids, evaporation, and condensation processes in closed metal objects by dynamic and static neutron radiography;

- investigation of apparatus such as automotive engines, fuel injection systems and, in general, hydraulic systems by quantitative neutron photogrammetry and dynamic neutron fluoroscopy; and

- study and quality control of thin-layered structures of solid and liquid surfaces by neutron reflectometry.

Neutron diffraction and SANS represent key techniques for the offshore sector, and are able to give, in a non-destructive manner, fundamental information in order to develop the design, manufacturing, and maintenance phases. In particular, while neutron diffraction is the only tool which allows the determination of the RS in the mass of the sample – including welded joints – a SANS-monitoring of the same joints may detect the cohesion breakdown that leads to internal surface growth and material fracture.

RS determination by neutron diffraction: theory and examples of industrial applications

The knowledge of spatial and directional RS distribution in a wide range of industrial plants is necessary in order to determine their influence on the material [1], which is a decisive factor for quality, life assessment, and safety. Stress concentration – in particular, tensile stress – should be avoided in order to increase fatigue life and avoid SCC and creep. Neutron diffraction almost certainly represents the most-complete method for the non-destructive stress measurement of components, and helps to reduce the conservative estimates which increase material and replacement costs. In the strain measurement, a collimated neutron beam (having a wavelength λ) is diffracted by a polycrystalline sample, and is then passed through a second collimator and reaches the detector. Both collimators slits define the investigated volume (see Fig.1), whose cross section, normally, can be as small as 1 x 1mm^2 or, in singular cases, smaller. The Bragg law allows determination of the interplanar distance d_{hkl}:

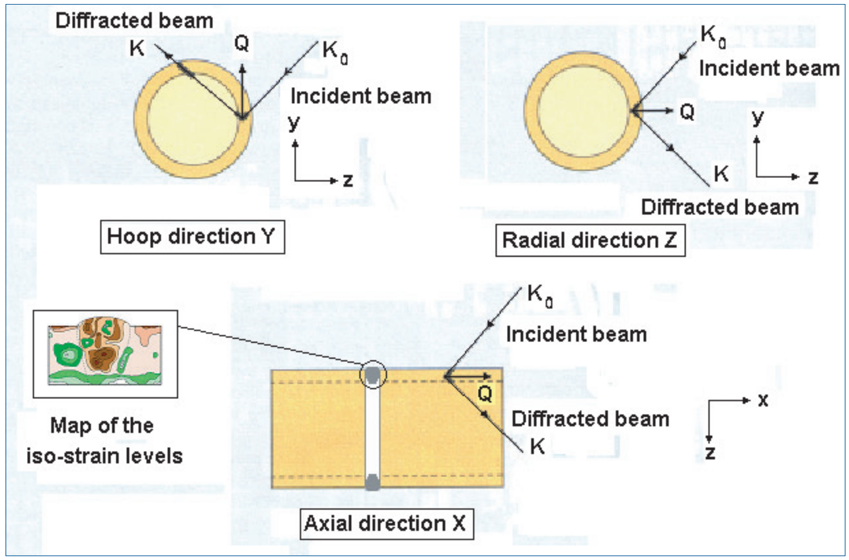


Fig.2. Geometry of neutron diffraction measurements in pipes in the main directions, showing iso-strain levels in the welded area and its surroundings.

$$n\lambda = 2d_{hkl}\sin\theta \quad (1)$$

where:

the integer n is the diffraction order,
2θ the take-off angle related to the maximum of the Bragg diffracted intensity peak, and
hkl are the Miller indices of the investigated lattice planes

The corresponding lattice strain is given by the relationship:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_0}{d_0} \quad (2)$$

where d_o is the hkl interplanar distance in a stress-free reference material [2].

The stress values can be obtained, consequently, by knowing the elastic constants of the material and using the relationship:

$$\sigma_{ii} = \varepsilon_{ii}\left(\frac{E(1-\nu)}{(1+\nu)(1-2\nu)}\right)_{hkl} + \left(\varepsilon_{jj} + \varepsilon_{kk}\right)\left(\frac{\nu E}{(1+\nu)(1-2\nu)}\right)_{hkl} \quad (3)$$

where i, j, k = 1, 2, 3, respectively. For a full treatment of the theoretical bases, see Refs 1-4.

The RS induced by welding can, as is known, compromise the structural integrity of a pipe which is, therefore, thermally processed in consequence. In this investigation, the RS of a 2.25Cr-1Mo ferritic arc-welded pipe after relaxation heat treatment was determined [4]: the dimensions of the welded pipe were:

outside diameter = 218mm
internal diameter = 178mm
total length = 355mm.

The temperature and pressure ranges were 350°-540°C and 100-200bar. The material that was used was preferred to austenitic steel due to its reduced problems of weldability. However, creep represents the main problem, as the manufacture, drawing, and welding processes induce RS. Measurements of the RS by neutron diffraction were performed before and after the heat treatment, and the pipe wall was investigated at the following depths: 2.5mm, 5mm, 7.5mm, and 10mm. Figure 2 illustrates the geometry of neutron diffraction in the main directions; the same figure shows how it is possible, by such technique, to map the iso-strain levels in a welded area and its surroundings. A clearly asymmetric progression of RS appeared across the weld: the values increased from one side of the weld zone to the other, following the direction of the weld pass. This trend can be attributed to the asymmetry of the welding process, initiating a higher tension in the fibre which was the last to cool in

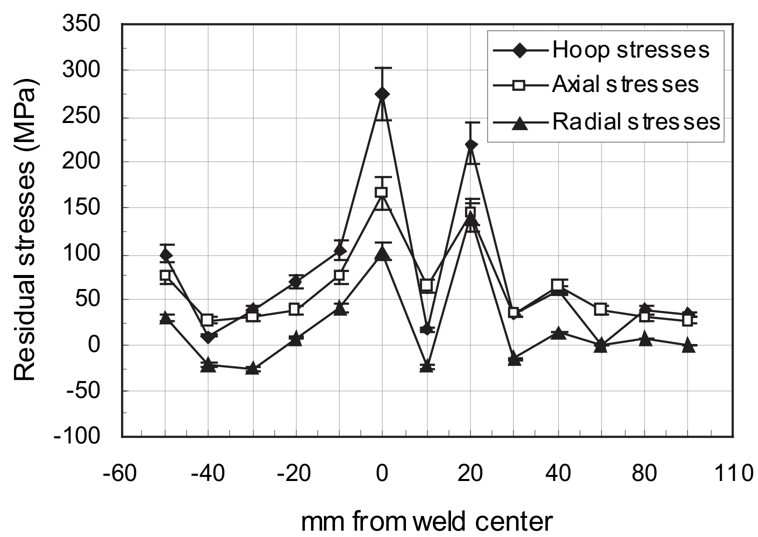
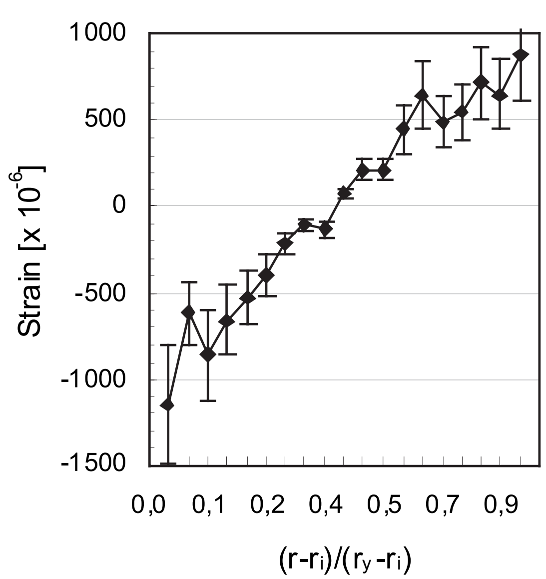


Fig.3. RS determination by neutron diffraction in a 2.25Cr-1Mo ferritic arc-welded pipe after heat treatment (5mm depth).

comparison with those in the contiguous regions. The RS after heat treatment (Fig.3, with reference to the 5-mm depth) appear to be zero in the radial direction, while in the axial one it is reduced by comparison with the stress before heat treatment, demonstrating a mono-dimensional status. The greatest tension appeared in the hoop direction, as a result of the cylindrical geometry. In the welding process, tension values up to 250MPa resulted (compared to 400MPa of tangential tension before heat treatment), from which a partial permanence of the initial manufacturing memory is shown.



RS measurements by neutron diffraction were performed in the pipe subjected to an internal pressure of 2500 bar, with the aim of inducing a strain in the material: the strain profile through the pipe was determined in the hoop direction, as shown in Fig.4.

A particular example showing the correlations between extrusion and quenching effects on the RS was given by two AA6082 extruded cylindrically-shaped samples (height = 50mm, diameter = 100mm) [5]. The same alloy is widely adopted in Europe for extruded components for structural applications, and it is quench sensitive: important properties, for example, can be reduced by too-slow cooling rates, while heavy distortion of the extruded section can be induced by too-fast cooling rates. The specimens were submitted, before the measurements, to an annealing process at high temperature; one was submitted to a low quenching rate, while the other to a high quenching rate. RS measurements were performed at points on radial straight lines perpendicular to the axis using a neutron beam section of 4 x 4mm^2. Three orthogonal directions of strain were considered: axial, radial, and hoop; these directions were assumed to be the principal ones. Figure 5 shows the stress values obtained (in GPa) and the geometry of the experiment.

Fig.4. Hoop strain in a CrMo steel alloy submitted to an internal pressure of 2500bar.

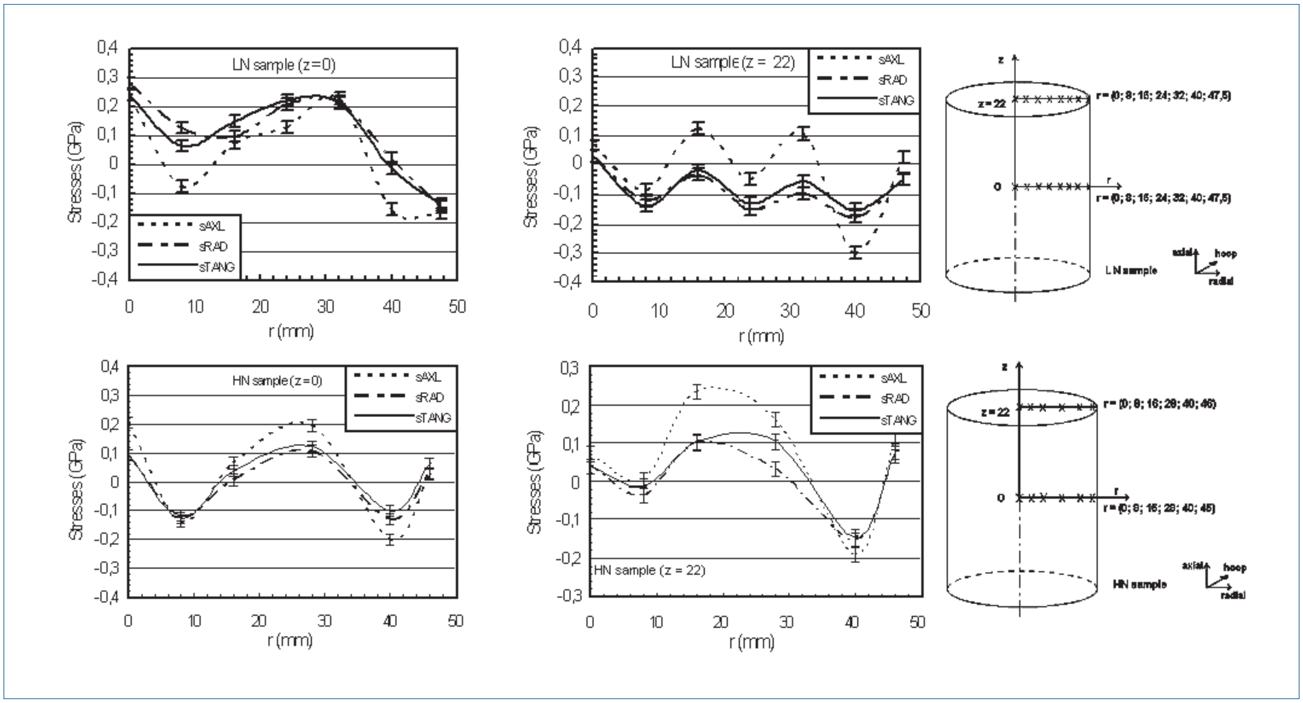


Fig.5. RS determination by neutron diffraction in AA 6082 extruded samples submitted to different quenching rates: stress data and geometry of the experiment (reprinted - with permission from Elsevier - from J.Alloys and Compounds*, 378/1-2, M.Rogante, P.Battistella, F.Rustichelli, 'Residual stress measurement by neutron diffraction in AA 6082 extruded samples submitted to different quenching rates', 335-338, 2004).*

Neutron diffraction was also used to map the strain surrounding two defects introduced into plates of pipeline-grade steel, one in the plate before uniaxial loading, and the second in the plate held at a constant background load that ensured the stress at the edge of the defect exceeded the elastic limit of the material [6].

A section of commercial X-70 pipeline was studied using neutron diffraction for the measurement of the RS, using x-ray diffraction to study its crystallographic texture, and by microstructural analysis. The results showed good correlation with the manufacturing steps applied for this type of pipe [7].

Intercrystalline and intergranular cracks, as is known, can be created in pipes and vessels as a consequence either of the construction technique, or of the presence of aggressive chemicals such as H_2S. Cracks of considerable importance can start in narrow welded zones of a particular hardness, and gradually increase from small sizes to the full width of the pipe wall (see Fig.6). Neutron diffraction can be successfully used in such cases to find the spatial and directional distribution of the internal RS before and after heat treatment.

Studying the microstructure by SANS: theoretical basis and practical examples

The SANS (small-angle neutron scattering) technique allows materials to be studied in the nanoscale range (10Å-1000Å), and has the advantages of being both non-destructive and providing information with high statistical precision, due to it averaging over a macroscopic sample volume. The specimen can be investigated at various times after more working or heat-treatment, and the small absorption of neutrons often allows measurements to be taken down to material thicknesses of only 1cm. Figure 7 shows a schematic of a SANS instrument: the neutron beam is monochromatized by a multi-

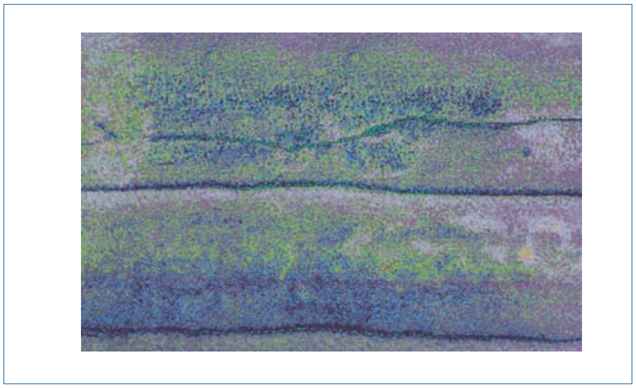


Fig.6. Traversing longitudinal crack in Fe 510.2KG/UNI.

disk velocity selector, and the wavelength distribution can be varied by changing the angle between the selector axis and the direction of the same beam. The beam intensity is monitored by a fission chamber, and formed by a collimator tube or a variable collimation path system, allowing optimization of the flux and resolution for different sample-to-detector distances. The scattered neutrons are detected by a 64 x 64 pixels two-dimensional position-sensitive detector with a pixel size of 1cm x 1cm, and software allows control and data acquisition. The primary data treatment includes the regrouping of the two-dimensional scatter patterns depending on the type of scatter (isotropic or anisotropic), background subtraction, correction for transmission, and normalization to standard samples.

In the SANS experiments, the scatter intensity I(q) was measured as a function of the scatter direction, if elastic scattering dominates the interaction between the neutron and the nuclei, and the vector q corresponds to the momentum transfer. The low limit ($qR_g < 1$) behavior of scatter intensity can be expressed by the Guinier relationship:

$$I(q) = \Phi K^2 V^2 \exp\left(-q^2 R_g^2\right) \quad (4)$$

where:

R_g is the radius of gyration of each individual scattered neutron,
K is the contrast,
V is the total volume, and
F is the scatter concentration.

R_g can be determined even if I(q) is known only in arbitrary units. In the large q region of scatter, the signal due to the interaction between each scattered neutron and the embedding medium dominates the intensity. This behavior can be represented by the Porod law:

$$I(q) = \left(2\pi K^2 \Sigma\right) q^{-4} \quad (5)$$

where σ is the total area of the interface per unit volume of sample. The limit of the Porod law is $qR > 4$; for a detailed treatment of the theoretical basis, see Refs 8-12.

The most important aspects of a materials' strength relate to welded metals, where the risk of fracture appears to be much higher than that in the base metal [13-16]. The higher risk of a joints' fracture results from (non-uniform) mechanical stresses and other factors due to ageing (thermal treatment, fatigue, corrosion)

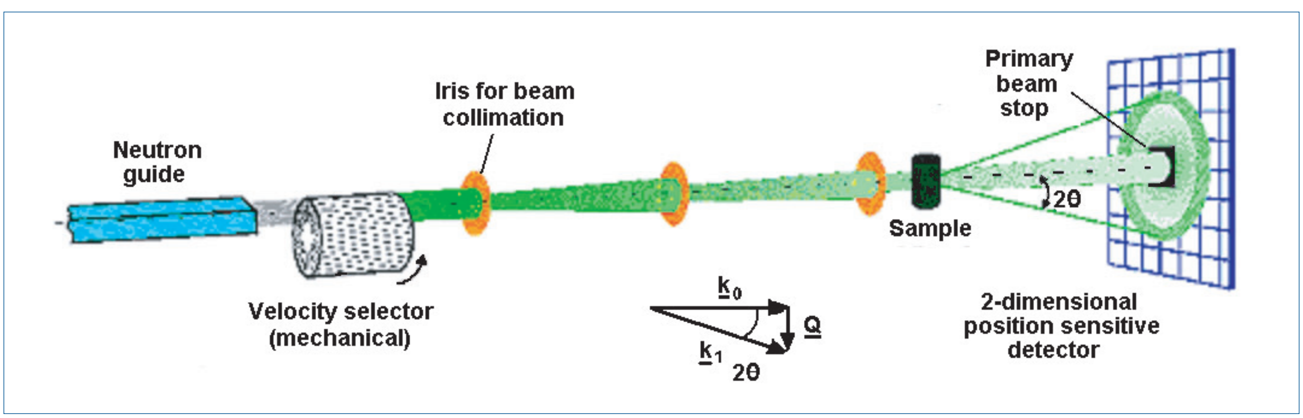


Fig.7. Schematic of the SANS instrument.

[14, 15]. Preventive examination of a material's nanostructure can identify degradation trends, in order to avoid fracture and predict the material lifetime. The information gained by diffraction studies (such as RS and micro-strains) can be completed by SANS, providing data on character, number, and size of defects by micro-beam scans detecting the nanopores (nanocracks) [17-19]. The physical basis of the fracture process is closely related to nanodefects: the investigation of porosity kinetics, dependent on various factors (including stresses, temperature, radiation), is therefore very important. Mechanical properties (creep resistance, joints ageing) are determined by the nanostructures (precipitates, pores, and dislocations groups) in the metal [13-16]. A non-uniform degradation of material leads to a fast development of cracks, critically reducing the lifetime of a joint.

A current project that is under way concerns the SANS investigation of P91 martensitic Inox steel (9Cr-1Mo-V-Nb) samples taken from 14-mm thick, 24-in diameter, pipe. The samples are longitudinally welded in the straight sections, and double-axially welded at the connections. The system is being submitted to 3000-8000 hours accelerated-creep tests, i.e. to 7000 start cycles after 100,000 hours (at a maximum gradient < 10°C/min); the test temperature is ~545-625°C, and the test pressure ~50bar. SANS analysis, with the variation of mechanical loading, can provide information on the nanoscopic and microscopic defect developments: from dislocations to voids and cracks. Thermal treatment produces some inclusions (precipitates), and it is also possible to identify their characteristics (such as number and size) by knowing their chemical composition (for instance, carbides Cr_6C_{23}, etc.). A sample from the original material will be investigated before the test, and other samples after the test, including the welded sections, in order to check the microstructural changes (nano-defects, voids, etc). The comparison of the SANS cross-sections for fresh-prepared and aged samples will give data on the size distribution and concentration of new defects induced by the mechanical treatment. The main aim of the creep tests is to determine, before reaching 30,000 hours, the accelerated creep test data which will help to predict the stress/strain level at 100,000 hours. Another aim is to perform an analysis of the following effects: temperature, stress and strain consequence (which is indispensable to correctly define the temperature), hold-time, acceptable creep type, and relative $d\varepsilon/dt$. The same equation will represent the strain as a function of temperature and stress, also taking into account the void changes after thermal treatment. The results are expected to represent very useful information for the pipeline industry.

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