Neutron characterization of ageing and creep processes in materials and parts from decommissioning of nuclear facilities

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Abstract

Safety conditions and lifetime extension of a Nuclear Facility (NF) are fundamental and a deep knowledge of ageing and creep processes linked with the involved materials and parts is essential to guarantee high levels of reliability and to face severe natural and plant-centred incidences. The analyses classically used in this case, involving non-destructive techniques (NDT) and in-service inspections required by the norms, can present a lack of information. The data acquired, consequently, need to be complemented. Decommissioning of NFs is an important chance to get materials and parts submitted for various years to ageing and degradation, consenting novel diagnostic activities beneficial to suggest eventual additional measures related to the installation of new components or in planning a postponed decommissioning, to increase safety and dependability. Neutron beam techniques (NBT) can be helpful to characterise, in a non-destructive and non-invasive way, materials and parts of nuclear/traditional interest. Positive recommendations exist to exploit neutron methods for reactor materials' control. In this paper, ageing processes occurring in the NFs sector are briefly introduced and the main neutron techniques are briefly described. Some examples of applications, then are presented. The results obtained by neutron analyses, linked with information on nature and features of materials' damages, assist to comprehend the trends of fracture and to evaluate a latent picture of preliminary degradation processes leading to any fast crash of material, including inoperability periods before the decommissioning activities.

Keywords: Nuclear Facility; Ageing; Creep; Neutron beam techniques.

Nükleer tesislerin devreden çıkarılmasından kaynaklanan malzeme ve parçalardaki yaşlanma ve sürünme süreçlerinin nötron karakterizasyonu

Öz

Bir Nükleer Tesisin (NF) güvenlik koşulları ve ömrünün uzatılması temeldir ve ilgili malzeme ve parçalarla

bağlantılı yaşlanma ve sürünme süreçleri hakkında derin bilgi, yüksek düzeyde güvenilirliği garanti etmek ve ciddi doğal ve bitki merkezli olaylarla yüzleşmek için gereklidir. Bu durumda klasik olarak kullanılan, tahribatsız teknikler (NDT) ve normların gerektirdiği hizmet içi denetimleri içeren analizler, bilgi eksikliği gösterebilir. Sonuç olarak, elde edilen verilerin tamamlanması gerekir. Nükleer Tesisin hizmetten çıkarılması, çeşitli yıllar boyunca yaşlanma ve bozulmaya maruz kalan malzeme ve parçaların alınması için önemli bir şanstır, yeni bileşenlerin kurulumuyla ilgili nihai ek önlemler önermek için faydalı olan yeni teşhis faaliyetlerine onay vermek veya güvenliği artırmak için ertelenmiş bir hizmetten çıkarmayı planlamak için faydalıdır. ve güvenilirlik. Nötron ışını teknikleri (NBT), nükleer/geleneksel ilgi konusu olan materyalleri ve parçaları tahribatsız ve istilacı olmayan bir şekilde karakterize etmeye yardımcı olabilir. Reaktör malzemelerinin kontrolü için nötron yöntemlerinden yararlanmak için olumlu öneriler mevcuttur. Bu yazıda sektörde meydana gelen yaşlanma süreçleri kısaca tanıtılmakta ve başlıca nötron teknikleri kısaca anlatılmaktadır. Daha sonra bazı uygulama örnekleri sunulmuştur. Nötron analizleriyle elde edilen, malzeme hasarlarının doğası ve özellikleri hakkındaki bilgilerle bağlantılı sonuçlar, kırılma eğilimlerinin anlaşılmasına yardımcı olur ve hizmetten çıkarma faaliyetlerinden önceki çalışamazlık dönemleri de dahil olmak üzere, malzemenin herhangi bir hızlı çökmesine yol açan ön bozunma süreçlerinin gizli bir resmini değerlendirmeye yardımcı olur.

Anahtar Kelimeler: Nükleer Tesis; yaşlanma; Sürünme; Nötron ışını teknikleri.

1. Introduction

Ageing and creep resistance, like other mechanical properties, depends to a great extent from the nano- and micro-structure formed in the metals. A non-uniform degradation can accelerate the progress of cracks favouring a serious reduction of the component's lifetime and therefore of the system [1]. Radioactive radiation and high operational temperature may produce substantial changes in the nano(micro)-structure of metallic components of NFs, e.g., piping, pumps of the primary circuit and related components, pressurizer, vapour generator and welds, plates, irradiated materials of the reactor vessel. Such changes can concern also non-metallic parts like those made of concrete for shielding, also varying their macroscopic characteristics. The mechanical properties that are crucial for safety and lifetime of NFs, consequently, can be weakened.

Lifetime extension, especially, is a major economic interest since the operational cost of a NF and in particular of a Nuclear Power Plant (NPP) is not significant in comparison with the investment cost. To extend lifetime it is crucial to get accurate information on structural alterations of the most important parts of the involved plant.

Classical methods to assess macroscopic properties and especially those mechanicals allow measuring the net sum effect of the microscopic variations. The damage created by the irradiation can be studied on model materials or on specimens belonging to real construction elements. Nuclear safety conditions also include monitoring of the materials' state (i.e., mechanical characteristics), by testing control samples of the same materials working in the identical regime, even taking larger forth-coming fluence, to provide an early observation of dangerous trends in the changes of materials' properties. To simulate such exposure, additional tests are carried out by irradiating materials and/or parts sensitive to damage from radiation and whose functional safety characteristics may be affected by exposure to radiation during exercise. Radiation aging, in this case, is performed on the sample under normal and abnormal operating conditions. This is comparable to what the component is undergoing during employment concerning the type of radiation and the exposure dose. These tests are performed also changing key parameters such as types of radiation, that can be applied separately, and exposure rate. The absence of effects other than those faced in the operating conditions, anyhow, must be always ensured. In addition, the temperature effects should be considered, which are part of the main factors producing ageing and creep. New machine learning platforms have been recently set up, to detect and quantify radiation-induced defects of parts and testing materials in nuclear reactors [2].

The measurements on model materials, anyway, need to be associated with complementary measurements on real structural materials. The irradiation damage, actually, strictly depends on various factors such as the adopted technologies and the concentration of alloys' components.

Traditional investigation methods involved by the existing norms for the NPP sector include also: mechanical tests to measure the alterations of macroscopic properties; X-rays; mechanized ultrasonic investigations concerning pre-service inspection; phased array ultrasonic testing. The achieved information, nevertheless, is not exhaustive and needs to be completed. In this context, NBT offer substantial advantage in comparison with the

classical analysis and fractography, helping to solve key problems associated with the methodological limitations of these methods [3, 4].

Decommissioning, defined as the cessation of operations and the withdrawal of the NF from service, is an exclusive opportunity to test really aged materials and parts, e.g. old NPPs with gas-cooled, graphite-moderated or boiling water reactors [5]. It is also a singular chance to exploit innovative diagnostics and perform actions in perspective of future installation of new components, aiming to increase security and consistency.

The risks during decommissioning are lesser with respect to the operating life of the NF, also since the process and the auxiliary systems are no more submitted to the stresses due to pressure and exercise conditions. Nevertheless, researches of accumulated defects in decommissioned materials and parts as dependent on duration and intensity of the irradiation (fluence) are very relevant. Neutron structural studies of ageing concerning materials submitted to irradiation, mechanical loading, high temperatures and chemical attacks, in this case, can give a fundamental support. Nano(micro)-defects such as cracks, dislocations, pores and vacancies, result from operative conditions (irradiation, mechanical and thermal stress). Knowledge of nature and features of these damages consents to understand the materials' fracture trends and to assess a latent picture of initial degradation processes that lead to any fast crash of material.

In decommissioning, several nuclear safety conditions requiring careful checks of the preservation state and structural checks of the components do not exist. Systems that are classified relevant for safety are very reduced in number and extension during the decommissioning and they tend to decrease during this phase. Nuclear safety conditions, anyhow, include the monitoring of the materials state by using the said tests with control samples. A main aim of the neutron investigation, in this case, is a subtle structural analysis of these samples used for monitoring, to find the same structural features like in broken or very aged decommissioned materials, which got very high radiation doses or undergone other actions damaging their structure. The possible damages due to neutron irradiation to the vessel's structures, e.g., are of high interest also during the decommissioning, when the vessel is without fuel and it is practically submitted to the only load of its own weight. The study of effects on structure in decommissioned reactor vessels, indeed, can find in detail the structural transformations of the involved material during a very long period of its exploitation, providing data to restore the kinetics of structural degradation and elaborating the criteria of durability of the existing reactor equipment. It should be considered also that the rather limited amount of economical budget, sometimes, does not consent to start the dismantlement of nuclear units without accumulation of sufficient financial resources. The time needed to build storage and disposals, thus, may result in choice of variant with long safe storage of units under surveillance [6].

Phenomena like thermal shock under pressure, due to the embrittlement of the vessel's materials during operation, may have a reduced importance in the case of decommissioning. However, a comparative study of structural changes in the original (fresh-prepared) materials and in the fragments of decommissioned materials can be very significant. With this aim, both kind of materials should be mechanically and/or thermally loaded to observe the embrittlement development induced by structural defects like nano-phase inclusions, which are clear visible in neutron scattering.

Safety authorities have not yet raised the issue of the systems integrity in terms that should require NBT. Nonetheless, the application of NBT for deep subtle structural analysis of reactor materials is in progress worldwide. The certification of cold neutrons scattering methods, as briefly described in Chapter 2, can be issued as a result of several experiments carried out and of highly developed data treatment methods. In this perspective, many positive recommendations exist from the International Atomic Energy Agency (IAEA) to use NBT for reactor materials control.

A main technological and scientific objective in the nuclear sector, conclusively, is to create a scientific background to elaborate widely used effective diagnostics methods to extend the life of reactor materials.

2. Neutron beam techniques

2.1. Neutrons

Neutrons are elementary particles nearly empty of electric charges, forming together with protons the atomic nucleus. They are not interacting electrically with electrons and nuclei in matter, thus they can penetrate the matter in depth. X-rays offer exceptional resolution but they are easily absorbed by materials, being able to penetrate only layers of their surface. The linear absorption coefficient of neutrons, instead, is ~ 1000 times weaker than X-rays, so they penetrate matter up to several centimetres, i.e. ~ 2 - 3 cm for steels and ~ 5 - 6 cm for aluminium and its alloys.

Neutrons are produced by continuous sources (nuclear reactors) or by pulsed sources (accelerators). In continuous sources, they are created by the fission reaction of heavy nuclei such as ^{235}U or ^{239}Pu , with very high initial level of energy. Then, they are slowed down to thermal energies by means of moderators. Finally, they are subjected to collisions that further slow them down to bring them into thermal equilibrium with the surrounding medium. This thermal equilibrium allows using the energy equipartition theorem as follows:

$$1/2 mv^2 = 3/2 kT \quad (1)$$

and also:

$$\lambda^2 = h^2/3mkT \quad (2)$$

where k is Boltzmann's constant, m , v , λ are respectively the mass, velocity and wavelength of the neutron, T is the temperature in degrees Kelvin.

The consequent distribution of neutrons as a function of velocities belongs to the Maxwellian type, where λ is in the range 1.55-1.33 Å for T in the range ~273-373 K (0÷100 °C).

Special guide channels transfer the produced neutrons from inside the core to the different instruments like diffractometers and spectrometers.

2.2. Small angle neutron scattering

Small angle neutron scattering (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 [3, 4, 7-10].

2.3. Neutron diffraction

Neutron diffraction (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 [3, 4, 11-13].

2.4. Prompt gamma activation analysis

Prompt gamma activation analysis (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 [3, 14, 15].

2.5. Neutron radiography

Neutron radiography (NR) allows 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. NR, e.g., allows visualising and assessing material's distribution within macroscopic volumes of cement's specimens, as well as defining water's movement through the investigated samples. NR images of structures are also helpful to validate conventional measurements. The theoretical bases of the NR technique can be found in various references, e.g. in [3, 16-18].

3. Examples of applications

Welding process is a major source of RS. Particularly in the heat affected zone (HAZ), it often generates large tensile RS reaching values roughly corresponding to the material's yield strength, balanced by minor compressive RS wherever else in the studied part. Contraction of molten weld metal all through solidification is hindered by adjacent colder metal, therefore generating RS. The major problems linked with welding involve also the formation of heterogeneities, typically inducing a lower chemical stability and a higher concentration of mechanical defects. The duration of parts submitted to very high temperature, cyclic loadings and/or erosion, hence, is decreased since creep, fatigue and wear damages in welded zones result enlarged. Intergranular and intercrystalline stress corrosion cracking (SCC), furthermore, can appear in narrow welded zones - e.g., in pipes and vessels -, due to RS generated during the manufacturing process and to possible occurrence of aggressive elements [19].

The global state of the art of welding project processes linked with nuclear/traditional industrial applications practically consists: in numerical models reproducing welding procedures; in primary interests in investigating welds to enhance their qualities. New welding project methods were studied based on FEM calibrated against conventional experimental results and on real key parameters' values obtained through nano(micro)-structural investigations of different welded joints by NBT, considering different metallic materials and welding processes [20].

Several analyses of welds have been performed, till now, showing the advantages of exploiting NBT to achieve essential data not available by using other means. SANS and ND allow analysing, e.g., massive and bi-metallic welds, weldment by friction welding process, TIG welds and repair welds, also investigating the effects of radiation on RS and defects' formation. NBT make possible also:

- to improve analytical methods to assess the total fatigue life of welds submitted to surface treatments and/or exposed to variable-amplitude loading, to find potential means to increase fatigue strength

- to analyse the control factors of fatigue behaviour related to welded components
- to apply probabilistic methods in order to assess potential nucleation of nano(micro)-cracks within a welded joint due to fatigue damage processes.

Knowledge of the inner and sub-surface RS distribution determined by ND in massive samples allows revealing the cause-effect unknown connections between the existing state of a given metallic part and the potential types of failure under operating conditions. It is possible, therefore, to estimate also the material resistance to crack propagation under operating conditions. A suitable failure predictive model e.g., requires the contribution of valid methods to collect and catalogue specimens, as well as of data analysis [21-23]. An example of RS assessment by ND in pipes concerns a 2.25 Cr1Mo ferritic arc welded pipe before and after relaxation HT [24]. Figure 1 reports the obtained hoop RS.

Figure 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 NFs can be performed concerning dismantled parts and new weldment made of the same constitutive materials. In weldment of austenitic steel pipes of NPPs, delayed reheat cracking can occur in the HAZ, needing repair if major cracking arises. Such repair welds influence the RS distribution, which is important to know in order to evaluate the effectiveness of the repair and to estimate the kinetics subsequent creep damage. ND, in this case, can supply crucial information. The greater risk of joints' fracture is due to the non-uniform mechanical stresses and to other factors producing ageing such as fatigue and thermal-related RS produced by HT. Notions of metal fracture and welded joints are currently mainly based on the use of optical and electron microscopy - especially, scanning electron microscopy (SEM) - to examine surface structures and defects in thin slices [25, 26]. Fracture surface areas and fractal properties of profiles and surfaces were examined as a part of different studies. The concept of fractals was developed underlining the different natural self-similar structures [27]. The two main classes of fractal structures are volume fractals (i.e., aggregated clusters of small particles) and surface fractals (i.e., systems with irregular interfaces and grain boundaries).

SANS allows verifying the fractal nature of structures and assessing the fractal dimensions [28]. This technique can provide crucial information on turbo-machinery parts and samples of constitutive materials, e.g., Inconel 738, Udimet 520 and Udimet 720. It is able to characterize precipitates in a non-destructive and non-invasive way [29-31], also dependently on temperature, hours of service and ageing time: thereby evaluating parameters largely responsible for the functional properties of these parts. Figure 2 reports the volume distributions of precipitates vs. size, for positions 1 and 9 of two turbine blades made of Inconel 738, one in the as-fabricated state and the other one after 25,000 hours of service, investigated by SANS by adopting a neutron beam cross-section of $3 \times 15 \text{ mm}^2$ [21].

Figure 2. Volume distributions of precipitates vs. size, for two turbine blades made of Inconel 738, one in the as-fabricated state and the other one after 25,000 hours of service, investigated by SANS [21].

Three populations of precipitates differing by characteristic size have been revealed, showing the larger differences along the scan in the blade after service.

To detect nano(micro)-structural alterations disjointedly in reactors materials of working NPPs, structure and precipitate morphology of vessel steel parts were investigated by SANS. Such parts are usually submitted to elevated neutron radiation and severe thermal conditions. Samples cut from 15Kh3MFA steel cracked specimens belonging to standard VVER-440 reactor materials were analysed, after an ageing HT at 295°C for 360 days. An anisotropic precipitate structure formed at the nanoscale level was revealed, due to macroscopic texture, together with the inhomogeneity of the vessel raw material [32].

An AISI 316L steel specimen was submitted to cyclic surface heating and simultaneously cooled by low pressure water flowing through channels across the specimen. To assess these RS by ND as a function of depth along a line from surface to central hole axis, thermo-mechanical RS were simulated, connected with the pulse nature of thermonuclear fusion reactor [33].

SANS technique was used to investigate base material and welded part of a 100 mm thickness sample cut out from a 08H18N10T austenite stainless steel plate. This sample was wire welded in electric arc and submitted to HT, cooling down in water from the initial temperature of 1050°C. The cross sections showed three fractions of point like defects, having a larger amount (by factor 5) in the base metal as compared to the welded part [20].

A feasibility study was performed for a SANS investigation of P91 martensitic steel (9Cr1MoVNb) specimens obtained by cutting 14 mm thick 24" pipes submitted to 3000÷8000 hours accelerated creep tests. These tests consisted in 7,000 start cycles after 100,000 hours with a max. gradient <10°C/min. The specimens contained longitudinal welds in straight parts and double axial welds in the connections. The test temperature ranged from ~545°C to ~625°C, while the test pressure was ~50bar. SANS allowed obtaining information on number and size distribution of some precipitates that were created by the HT, by knowing their chemical nature (e.g., Cr₆C₂₃ carbides). The SANS investigation was considered before and after the test, to study nano(micro)-structural changes, e.g., nano-defects and voids. The main purposes of these creep tests were:

- to determine, before reaching the 30,000 operative hours, the accelerated data that facilitate the estimate of the stress/strain level at 100,000 hours
- to support the creation of constitutive equations representing the strain as a function of temperature and RS, also taking into account the voids' changes after HT; these equations should allow performing interpretative analyses of effects such as temperature, strain and RS, as a correct step to study the relations with primary/secondary phases of the creep [28].

Austenitic stainless steel with the GOST mark 08X18H10T is adopted, e.g., to built reactor internals, collectors of parogenerators and loop pipelines of the primary circuit. Maintenance and repair of primary circuit components involve high nickel weld filler. RS determination by ND was carried out on NPPs construction parts made of austenitic stainless steel coated with high-nickel alloy: the main aims were to prove the mechanical

analysis and to verify RS determination on welded material by numerical computer welding simulation. Measurements were performed on 25 and 15 mm thick samples, obtaining normal and transverse RS components [34].

NBT can contribute to study other materials and parts from NFs decommissioning, including nuclear waste treatment. Study of dense cements, ceramics and related materials, e.g., cement stones, is significant to create novel advanced components for nuclear-safety related structures with high functional properties (such as ageing resistance, cracks' formation, hardness, durability, stability of mechanical modules and ecological criteria). These high-performance materials can be considered as constitutive of corrosion resistive coatings for nuclear waste containers. Knowledge of their thermo-physical properties in broad temperature and moisture ranges is important for a realistic modelling of potential accidents in NPPs [35]. Conventional investigations of cements, including those for nuclear industry, use standard methodologies to acquire information on aspects primarily related to micro-structure, fatigue behaviour and computational mechanics. These investigations rely essentially on standard tests, theoretical studies and simulation-based analyses. Porosity, e.g., is a main parameter for cement, affecting mechanical and heat-insulation features. This parameter is generally assessed through gravimetric techniques, gas and liquid porosimetry (e.g., mercury intrusion porosimetry) and acoustic methods [36]. A campaign of various SANS investigations was carried out by the Rogante Engineering Office on polymer cement concretes made of Portland cement with added nano Al_2O_3 (γ type) mixed with PAV 22 polymer, produced by the Institute of Material Sciences and Metallurgy of the Ural Federal University, Yekaterinburg, Russian Federation. Substantial data, concerning in particular the size distribution of nano-sized pores, were obtained on basic parameters connected with degradation, fracture and other phenomena. These results can help to understand the structural basis for the physico-chemical properties and therefore to improve quality and durability of the considered materials, allowing a more reliable lifetime assessments. They can also contribute to optimise the material's consistency, the design of structural elements operating conditions and of the procedures supporting ecological criteria and enhancing quality and safety levels [37-40].

Different other neutron studies were performed concerning radiation damages in aged materials of NFs, e.g. [41-44]. SANS technique was used to investigate the nanostructure of SAV-1aluminum alloy, as constructional material of a WWR-M reactor core such as reactor vessel and support grid. An original sample and a sample-witness with accumulated the high dose $2.25 \cdot 10^{21}$ n/cm² of fast neutrons in reactor zone were analysed. As a result, the presence was detected of Mg_2Si and Si nano-phase inclusions having gyration radii $R_G \sim 3\text{-}40$ nm. The irradiation caused a decrease of the volume fraction of large entities and the content increase of smaller particles (size $\sim 8; 20$ nm), as a fragmentation of large entities under irradiation, since the total area of particles surface was unchanged [44]. It was also observed a qualitative change of defects which induced neutron scattering, as shown in Figure 3.

Figure 3. SANS data for pristine and irradiated SAV-1aluminum alloy.

It was a surprise that the irradiation produced a decrease of the scattering intensity, despite the active formation of various defects in the specimen, i.e. point, liner such as dislocations and their groups. In double log scales, the scattering intensities data demonstrate different slopes for the pristine and irradiated specimen, as shown in Figure 4.

Figure 4. Fractal behaviours of scattering intensities in pristine and irradiated specimen (1,2).

The imperfect borders of grains are initially visible in the scattering, when the intensity obey the fractal law:

$$I(q) \sim 1/q^\beta \quad (3)$$

with the parameter $\beta = 3.31 \pm 0.04$. This magnitude $3 < \beta < 4$ means that the grains surface has a fractal dimension $D_s = 6 - \beta = 2.69 \pm 0.04$. Such a value of D_s indicates a very imperfect border of grains, which result strongly coupled. The irradiation, however, makes a lot of low-dimensional defects such as dislocations. This leads to observe a lower slope of the fitting line when the intensity is described by the function:

$$I(q) \sim 1/q^{D_f} \quad (4)$$

with the fractal index $D_f = 2.56 \pm 0.06$ (mass fractal). The formation of a great number of dislocations dominating in scattering, consequently, was observed.

The final stage of a NF life cycle can include mechanical and hot cutting processes such as plasma torch and oxyacetylene arc metal. Analyses were performed on pipes and plates exposed to the reactor coolant steam, considering by use contaminated components below the free release level of $1 \cdot 10^4$ Bq/m², not yet contemplating radioactivity. Such study allowed estimating accurately: chemical and physical characteristics of the emissions produced during hot cutting tests; technical parameters such as cutting time and cutting rate vs. pipe diameter or plate thickness. The results focused on comparing the effects of plasma and oxyacetylene cutting processes and the chemical composition of the dusts collected by filtering the gaseous emissions [45–49]. SANS was used to investigate Fluoropore™ membrane filters, i.e. hydrophobic polytetrafluoroethylene membrane bonded to a high density polyethylene support, having a thickness of 150 μm, a pore size of 1 μm and a 85% porosity. These filters were used to capture the gaseous emissions. The distribution of inhomogeneity forms was evaluated in the range $10^0 \div 10^4$ Å, achieving information complementary to those obtained by transmission electron microscopy (TEM). While SANS supplied data on nano(micro)-structural characteristics averaged on a volume in the range between 1 mm³ and 1 cm³, TEM referred to very limited surfaces [50].

4. Conclusions

Safety of NFs and the need to eliminate any possible technological risk makes the most real a development of methods to forecast exploitation resources of nuclear installations.

The NF decommissioning activity is a real opportunity to carry out important comparative studies in depth of nano- and micro-structural features and difectoscopy of aged parts exploited for very long periods (in practice > 20-30 years) and fresh prepared samples, in order to found relationships between defects' characteristics and macroscopic functional properties of materials.

This is a key factor especially to progress new NFs components and their constitutive materials, contributing in the assessment of their suitability, since the more deep understanding of their behaviour.

Neutron techniques represent a valuable advance, helping to better study ageing and creep processes and to develop the knowledge of various safety-related properties and other important features of the involved materials.

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