

RADIATION RESISTANCE OF ELASTOMERIC AND LUBRICATING MATERIALS IN THE TRIGA MARK II REACTOR MIXED NEUTRON AND GAMMA IRRADIATION FACILITY

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ABSTRACT

Intense neutron fields induce substantial modifications of the physical and mechanical properties of polymeric materials. Protocols for irradiation and testing of elastomeric O-rings and lubricating greases have been developed, using the in-core irradiation facility of the TRIGA Mark II reactor of the University of Pavia. Four elastomers irradiated up to 2 MGy of absorbed dose were submitted to standard tensile test and compression set test. A remarkable increase of stiffness and brittleness is reported for some of the materials, while others feature more stable behaviours. Consistency was measured for seven different greases irradiated up to 5 MGy of absorbed dose. Outstanding out-of-scale grease softening is reported for some products. Some products rated as radiation resistant to pure gamma radiation feature more severe degradation in nuclear reactor irradiation conditions. These results present an original set of data achieved in mixed neutron and gamma radiation fields, generally very scarce in the literature.

1. Introduction

Materials used in the presence of intense neutron and gamma fields may suffer from radiation-induced modification of physical and mechanical properties. Among the non-metallic materials, the polymeric ones are known to be the most sensitive to ionising radiation [1]. Despite their well-known sensitivity to radiation, polymeric components are necessarily included in the design of nuclear facilities where intense neutron fields are produced. The most critical categories of components are represented by elastomeric O-rings, lubricating greases and oils, cable insulators, optical fibers, etc. Their use in such highly radioactive environments represents a largely unexplored challenge.

In the last decades, the subject of the radiation-induced materials damage was extensively investigated in the context of nuclear reactor technology, nuclear physics research and accelerator technology as well as in space applications. Among all, one of the most remarkable references is represented by the Yellow Reports produced by CERN laboratories, generally considered as a golden standard in accelerator technology and experimental nuclear physics (see [2] and references therein). Several categories of non-metallic materials have been irradiated and subjected to post-irradiation examination (PIE) [3]. Since it is easier and cheaper to irradiate polymers using gamma radiation than neutron

sources, most of the existing radiation damage data were produced using gamma radiation only, under the assumption that all different types of radiation cause equivalent damage to polymeric materials at equivalent absorbed dose. As far as organic materials are concerned, this *equal dose - equal damage* assumption has been widely accepted by the scientific and technological community for a long time, despite the lack of experimental evidences [4] [5]. For the mentioned reasons, neutron damage data on organic materials are lacking in the literature and comparative studies using different radiation sources are scarce [6].

Contrary to this widely accepted assumption, several documents supporting the need for further investigations on neutron damage data can be found [1] [7] [8] [9]. Reasons why the effects of mixed neutron and gamma reactor radiation in polymeric materials might differ from that of gamma alone are reported, along with a need for data comparing the effects of neutrons and gamma radiations.

To study the effects of neutron-induced damage compared to the more commonly studied gamma-induced damage mechanisms, selected elastomers and lubricating greases were irradiated by mixed spectrum of neutrons and gammas in the TRIGA Mark II reactor at the University of Pavia. This paper presents the results of post irradiation examination of these irradiated material specimens. Apart from the scientific relevance, the knowledge obtained from the performed PIEs provided useful inputs in materials selection and lifetime definition to the European Spallation Source (ESS), under construction in Lund, Sweden and the SPES facility, under construction at the Legnaro National Laboratories (LNL) of the Italian Istituto Nazionale di Fisica Nucleare (INFN) [10] [11]. Different protocols for irradiation and testing of categories of polymeric materials were developed and validated.

2. Materials Selection

Four elastomeric materials used for vacuum O-rings construction and seven lubricating greases were selected for the irradiation tests. The products represent a broad selection of typologies. Some of them have a radiation resistance declaration provided by the producer. However, producer declarations usually consider only the total amount of absorbed dose as a relevant parameter and refer to tests performed using gamma radiation only. Moreover, testing conditions and usability thresholds are usually not detailed. Some of the products were selected to fit the requirements for the use in the ESS and SPES facilities.

2.1 Elastomeric materials for vacuum O-rings production

One FPM fluoroelastomer (Viton®) and three EPDM based elastomers selected for irradiation and testing are listed in Table 1, along with a qualitative evaluation of their overall mechanical performance. The radiation resistance reported refers to the producer declaration. The selected products are used for the production of vacuum O-rings and are currently available on the market. FPM based elastomers are commonly used for this kind of application because of their excellent performance and resistance to temperature [12]. EPDM based elastomers are generally considered more resistant to gamma radiation than FPM compounds [3]. Nevertheless, the role of EPDM products as best choice for application in nuclear radiation environment should be questioned in the light of the neutron dosimetry that is highly influenced by the hydrogen content in the polymer blend. More information about the selected elastomers can be found in [13].

Tab. 1 : Elastomers selected to be tested in reactor mixed fields.

Product	Producer	Curing	Mechanical properties	Radiation resistance
VITON	Generic FPM		Good	Not declared
EPDM1	Generic EPDM	Sulfur	Fair	Not declared
EPDM2	EPDM 70 Perox Dichtomatik	Peroxide	Good	Not declared
EPDM3	Shieldseal® 663 James Walker	Peroxide	Very good	1.6 MGy dose gamma radiation

2.2 Lubricant greases

Seven lubricating greases selected for irradiation tests are listed in Table 2. Consistency, being the most important property used to characterise lubricating greases, is reported in NLGI (American National Lubricating Grease Institute) grade. Grease classification according to NLGI scale is universally accepted and most of the products are chosen on the base of this parameter only [14]. Some of the selected products are suitable for high-vacuum, high load and extreme pressure application, while others are general purpose. Radiation resistance declarations, when reported by the producer, refer to gamma irradiations only.

Tab. 2 : Lubricating greases selected to be tested in reactor mixed fields.

Producer and product	Base polymer	Thickener	Consistency	Radiation resistance
Lubcon Grizzlygrease No.1	Mineral oil	Li/Ca special soap	NLGI 0	1.2 MGy gamma
Kluber Petamo GHY 133 N	Mineral oil	Polyurea	NLGI 2	Not declared
Moresco RG-42R-1	Polyphenyl ether	Polycarbonates, silica add.	NLGI 1	15 MGy gamma
Lubcon Turmopolgrease 2	Polyglycol	Li soap	NLGI 2	Not declared
Kluber Klüberlub BE 41-542	Mineral oil	Special Li soap	NLGI 2	Not declared
Schaeffler FAG Arcanol Load 220	Mineral oil	Mixed	NLGI 1-2	Not declared
THK AFB-LF	Mineral oil	Li based	NLGI 2	Not declared

3. Experimental testing protocol and results

Specific irradiation set-ups and post-irradiation examination protocols were designed, developed and validated for elastomeric O-ring slices [13] and grease samples. Materials are irradiated using the Central Thimble facility of the TRIGA Mark II research reactor of the University of Pavia. Post-irradiation examinations are complicated by the residual activity of the irradiated materials, which must be handled by qualified operators. Neutron Activation Analysis (NAA) routinely performed on every product evidences, for example, the presence of ^{65}Zn in traces. Since it is a long-lifetime isotope, irradiated materials must be tested inside the nuclear reactor laboratory. As another constraint, only very small materials samples fit the limited volume available for irradiation in the in-core facility.

The realisation of a working, suitable and safe irradiation set-up for greases was more complicated than the one for O-rings because of the material semi-solid consistency. Grease handling is less efficient and leads more frequently to contaminations. Several preliminary tests were performed on small material samples to properly design the testing set-up and verify possible problematic radiation-induced effects. The relevant radiation-induced gas development promotes high grease mobility, making the material containment problematic. Acid gases are in some cases released, with evident corrosions of the surrounding environment. The final configuration for grease irradiation consists of a plastic syringe in which about 8 mL of material are uniformly distributed on its inner surface.

3.1 Irradiation conditions and dosimetry calculations

The Central Thimble is a 3.8 cm diameter aluminum pipe reaching the very centre of the reactor core (see Figure 1). The temperature inside the facility when the reactor is operating ranges between 60°C and 70°C. For the present study, the reactor was working at a power

of 250 kW. In this condition, the neutron flux in the irradiation position is $1.72 \cdot 10^{13}$ neutron/(cm²s). The flux of fast neutrons, having energy higher than 0.5 MeV, is $3.80 \cdot 10^{12}$ neutron/(cm²s). The thermal flux component of the spectrum is comparable to the fast one [15]. The gamma spectrum is a typical fission spectrum.

Dosimetry calculations in the Central Thimble are achieved using a model of the reactor realized with the Monte Carlo code MCNP5, developed for radiation transport (see Figure 1) [16] [17]. The absorbed dose in a mixed field highly depends on the material composition. The main mechanism of energy release in polymeric materials is usually represented by elastic scattering of fast neutrons on light nuclei. The average energy transfer is maximum when scattering occurs on a hydrogen atom. Secondary protons ejected in this process are expected to represent the most important contribution to the total dose [18]. For this reason, the neutron dose exhibits a direct correlation with the hydrogen content. To better simulate the neutron dose contribution, the main elements constituting the tested products are quantified via CHN analysis, allowing the carbon, hydrogen and nitrogen content to be determined in mass percentage. The remaining mass is modeled on the base of the producer declarations and on assumptions based on the polymer chemical nature of the material. Neutron and photon dose rate contributions are simulated using a specific material model for each of the selected products.

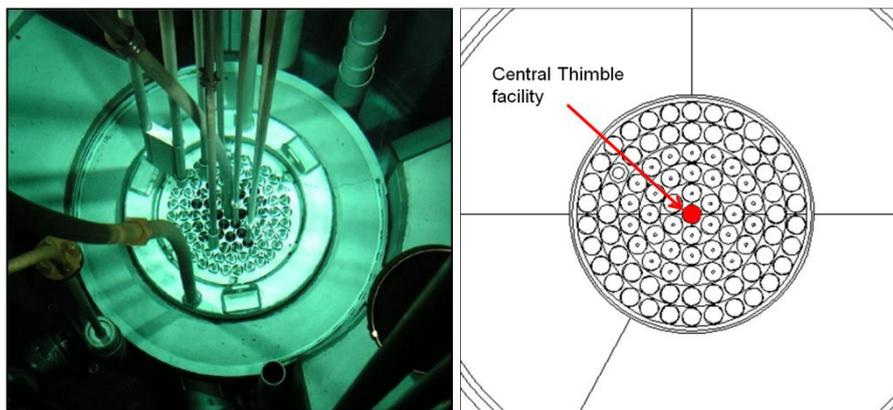


Figure 1. Left: a picture of the core of the TRIGA Mark II nuclear research reactor of the University of Pavia. The aluminum pipe of the Central Thimble facility reaches the middle of the reactor core. Right: a horizontal view of the reactor core as modeled in MCNP5. The position of the Central Thimble facility is evidenced.

Concerning elastomeric products, the hydrogen content in the selected EPDM ranges from 7.21% to 8.13% in mass percentage, while for Viton it amounts to 1.17% only. Total dose rates in the Central Thimble are about 0.68 MGy/h and 0.35 MGy/h for EPDM and FPM respectively. Gamma doses delivered to FPM and EPDM are comparable, being roughly irrespective of the specific composition for this kind of materials. By contrast, the neutron dose is about four times higher for EPDM, due to the higher hydrogen content. The neutron dose contribution is dominant in EPDM, representing about 65% of the total dose. The gamma dose contribution dominates in FPM, being 67% of the total. More details about dosimetry and about materials composition can be found in [13].

The hydrogen content in the selected greases ranges from 9.68% to 14.13% in mass percentage. Total dose rates range from 0.76 MGy/h to 0.94 MGy/h accordingly. For all the materials the neutron dose component is dominant, ranging from 66% to 71% of the total dose. Gamma contributions are comparable. The error associated to the calculated doses depends on the error on the measured fluxes in the irradiation facility, corresponding to 10%. According to these dosimetry consideration, radiation resistance declarations, usually based on gamma irradiation only, should be reconsidered in view of the material composition as well, strongly affecting the neutron dosimetry. The neutron dose absorbed by a hydrogen-free material is indeed several times lower compared to the one absorbed by a highly hydrogenated one. Fluorinated materials are included in the selection of the tested materials in view of their lower hydrogen content, making them more transparent to fast neutrons.

Dosimetry considerations play an important role in the evaluation of the best material to be used in a nuclear environment.

Radiation damage on non-metallic materials is influenced by several factors, such as type of radiation field used, dose rate, presence and penetration of the oxygen in the bulk material, sample geometry, temperature. Moreover, many of these effects act in a synergic way in assessing material damage [6][19][20]. For this reason, the results reported in the present study refer to the specific irradiation conditions of the Central Thimble. Prediction of materials behaviours in different irradiation conditions based on these results cannot be done in straightforward way. The predictive capability of the results obtained in this study for some target applications is currently under investigation [13].

3.2 Results for elastomeric materials

Samples of tested elastomers are irradiated at four dose levels in the specified irradiation conditions, corresponding to 0.5 h, 1 h, 2 h and 3 h of reactor time. Some of the most relevant mechanical and physical properties are measured for non-irradiated and irradiated samples to assess their evolution as a function of the dose. Among the mechanical properties, elongation at break, elastic modulus and tensile strength are measured using a standard tensile testing machine according to ASTM D1414. In the test, O-ring samples are elongated up to their fracture point.

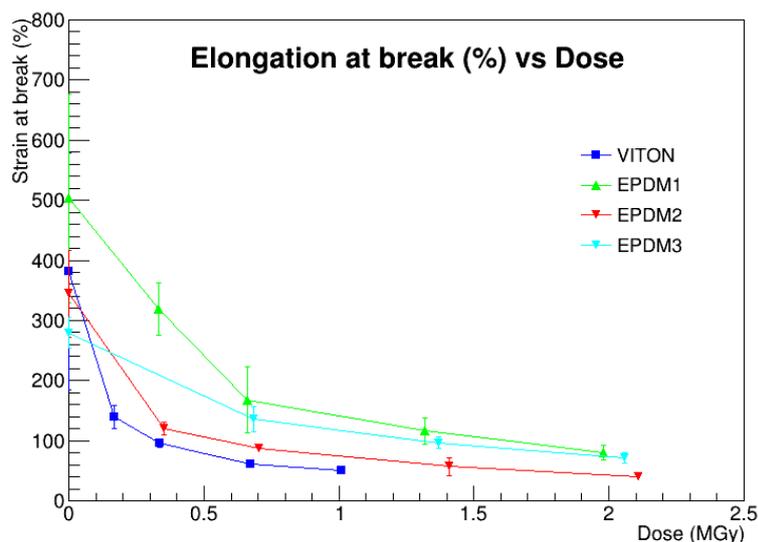


Figure 2: Elongation at break as a function of the absorbed dose for the four selected elastomeric materials used for O-ring construction.

Results of Elongation at break tests are reported in Figure 2 for the four materials. Important variations are measured for this property as a function of the absorbed dose. Since elastomeric materials are by definition able to elongate at least 100% without breaking, variations in this parameter below this absolute value should be considered as critical in the definition of radiation resistance. Elongation at break decreases sharply with absorbed dose for Viton, dropping below 100% at 0.3 MGy, starting from an initial average value of 400%. For EPDM 1 and EPDM 2 the value drops below 100% at 1.5 MGy and 0.7 MGy, starting from an initial value of about 500% and 350% respectively. For EPDM3, elongation at break value drops below 100% at 1.5 MGy. Considering its initial value of about 280%, this product features comparatively the best performance relative to the non-irradiated material.

Compression set is measured according to ASTM D395-03 and D1414-94. Results for the four materials are reported in Figure 3 as a function of the dose. Elastomeric samples are irradiated in an uncompressed state and are subjected to standard compression set test after irradiation. The diameter of the O-ring sample is subjected to a 25% squeeze during a 24 h long ageing at 100°C of temperature. The compression set is considered as a reliable

indicator of a permanent material deformation after a prolonged strain application. Its value ranges from 100% to 0%, the higher the value, the lower the elastic ability of the material in recovering its original diameter shape after the test. Compression set for Viton rapidly worsen up to 20% with absorbed dose. A brittle failure is reported at the same value. EPDM 1 exhibits poor initial compression set value that improves with the absorbed dose. EPDM 2 and EPDM 3 exhibit an almost stable compression set value as a function of the dose, featuring the best reported performance comparatively. A complete description of the results can be found in [13].

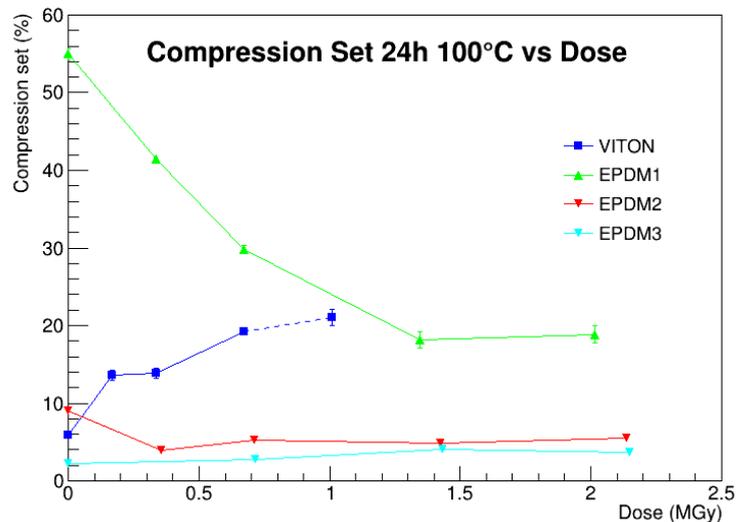


Figure 3: Compression set as a function of the absorbed dose for the four selected elastomers. The dotted line in the VITON graph indicates a brittle fracture of the specimen.

In addition, the following investigations on physical properties were performed: DSC calorimetry, IR analysis, DMTA analysis, swelling test and density measurement, aiming to correlate the evolution of the mechanical properties with the evolution of the polymer structure at a microscopic scale. The results of these tests, along with other mechanical results obtained in the study and not reported in the present paper, allow the following conclusion to be drawn.

Viton and EPDM1 exhibit the largest modifications of the tested mechanical properties as a function of the dose. In particular, Viton experiences a dramatic stiffness and brittleness increase over 0.26 MGy. Since the stability of the mechanical properties as a function of the dose is an indicator of radiation resistance, these materials can be considered as sensitive to radiation in the described irradiation conditions. It is interesting to note that the three EPDM-based materials display very different behaviour as a function of the dose despite their similar base polymer chemical composition. EPDM2 and EPDM3 display the best stability of the analysed mechanical properties, apart from elongation at break. EPDM3 is specifically designed for application in nuclear environment and is tested to gamma radiation up to 1.6 MGy of dose. It features, indeed, the most stable behaviour in the tested irradiation conditions. All the elastomers show that radiations promote a material stiffness increase and a progressive O-ring embrittlement. The outcomes of the chemical and physical analyses suggest that these phenomena are due to an increased cross linking of the polymeric chains constituting the material, as the predominant effect of radiation. Further investigations in this sense are ongoing.

3.3 Results for lubricant greases

Seven greases are irradiated at different dose values. The following irradiation times in the reactor facility are chosen: 10 minutes, 30 minutes, 1 hour, 2 hours, 5 hours, corresponding to dose values up to about 4.5 MGy. Only the most stable products were selected for five hour long irradiations. Greases are multi-phase systems originating from the dispersion of a thickener in liquid oil. The property most commonly used to characterise the grease quality is

consistency, being an indicator of the resistance to movement under stress. Consistency is measured for irradiated and non-irradiated samples according to standard ASTM 1403. To measure consistency, a penetrometer is used, whose cone penetrates under its weight into the flat surface of the material (see Figure 4, right). Penetration results are associated to consistency NLGI class values ranging from 000 (softer grease) to 6 (harder grease) [14]. Consistency is expected to exhibit a complex behaviour as a function of the absorbed dose, depending on the grease multi-phase nature. The first expected radiation-induced effect is the damage of the gelling structure of the thickener, being the most radiation-sensitive component of the grease. At higher dose values, a grease hardening can occur due to the radiation-induced viscosity increase of the base oil component [1].



Figure 4. Grizzlygrease No.1 irradiated at 0.14 MGy (left) and 0.42 MGy (center). The grease became almost fluid after irradiation (centre). Klüberlub BE 41-542 grease irradiated at 4.39 MGy, becoming almost fluid (right). The grease drips from the instrument. Consistency is measured using a penetrometer.

Consistency variations relative to the non-irradiated material are reported in Figure 5 as a function of the total absorbed dose for the selected greases. The error associated to consistency values, being lower than 5%, is based on repeated measurement performed on non-irradiated greases. A 10% consistency variation generally corresponds to a NLGI class variation, indicating an important radiation-induced modification of the most relevant mechanical property of the material. In this study, consistency class stability is considered as the feature characterising the materials radiation-resistance.

Five of the tested greases experience extreme consistency modifications, up to values exceeding the maximum range of the instrument. The material in these conditions is qualitatively completely different from the non-irradiated one. Under the force of gravity grease should remain in place as a solid body [14]. By contrast, some of the irradiated greases become almost fluid and drip from the testing instruments (see Figure 4). Fluid consistency is indicated by dotted lines in Figure 5. This radiation-induced modification is more severe than class variation and marks a dramatic evolution of the mechanical properties of the material. Remarkable variations are reported for Turmopolgrease2, Grizzlygrease No.1 and AFB-LF products for which NLGI class variation is reported at 0.13 MGy, 0.42 MGy and 0.94 MGy respectively. These three products feature the worst performance in the radiation-stability point of view comparatively, experiencing noticeable softening at dose values lower than 1 MGy. Klüberlub BE 41-542 and Arcanol Load 220 exhibit an almost progressive consistency increase as a function of the dose. NLGI class variation is reached at 1.75 MGy and 1.83 MGy respectively.

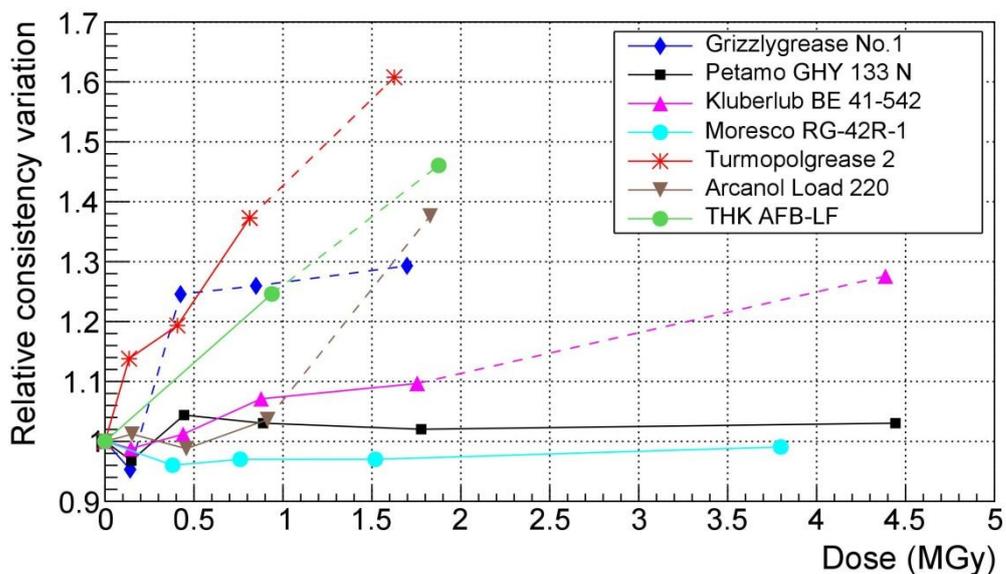


Figure 5. Relative consistency variation for the selected lubricating greases as a function of the absorbed dose. Dotted lines are used to indicate values rising above the maximum instrument range, corresponding to an almost fluid consistency. High values correspond to softer grease consistency and lower NLGI consistency class.

Moresco RG-42R-1 and Petamo GHY 133 N remain in their original NLGI consistency class up to the maximum investigated dose values of 3.80 MGy and 4.44 MGy respectively. These products feature the best radiation-resistance among the selected ones. Moresco product is declared by the producer to be radiation resistant up to 15 MGy of gamma dose, while Petamo has no radiation resistance declaration. The colour of some of the tested greases darkens by irradiation, evidencing chemical modifications. This phenomenon is particularly evident for Petamo grease, whose colour turns from yellowish (non-irradiated) to complete black at 0.44 MGy. Greases can contain special additives whose function is to prevent premature ageing or material oxidation. According to some producer declarations, colour change can be an indicator that additives are properly working and lubricating performance is not affected in case of colour modifications [21].

The results achieved on Grizzlygrease No.1 are particularly intriguing because its consistency abruptly increases after 0.42 MGy only, despite its radiation-resistance declaration up to 1.2 MGy of gamma dose. This difference can be attributed to the different irradiation conditions, for example the presence of neutron radiation and/or to the effect of a much higher dose rate.

According to the literature, grease radiation stability should be predominantly determined by the base oil chemical composition. For example, polyphenylether based products are known to be more radiation-resistant than mineral oil based one [1]. The outstanding stability of the Moresco product seems to comply with this consideration. However, since in many cases a radiation-induced fluidization of the material is observed, the thickener appears to be the most sensitive grease component. Its role in assessing the radiation-resistance of the material is relevant and as a consequence, radiation stability is more likely due to both the chemical nature on the base oil and the thickener one, along with their interaction at the microscopic scale. Specific additives used for the blend production are expected to influence radiation stability as well. The unexpected degradation of the Grizzlygrease No.1 product suggests that irradiation parameters play a relevant role in assessing radiation damage. For this reason, they should be specified in the radiation resistance declarations. The efficiency of fission neutrons in damaging this material can be higher than the pure gamma one. Generally speaking, it is very difficult to formulate hypothesis about the radiation-induced modification at the microscopic level based on results of penetration test only. Greases are

extremely complicated multi-phase systems, whose chemical and physical characteristics are difficult to be modeled and understood. Moreover, despite its effectiveness, the penetration measurement is not able to investigate the viscoelastic nature of the material. The lack of basic research into the fundamentals of grease in the academic community supports the need for further investigation [14].

4. Conclusions and further developments

Experimental studies of radiation resistance in reactor mixed fields on non-metallic materials for nuclear applications are ongoing. Specific protocols for elastomeric O-rings and lubricating greases irradiation and testing were developed and successfully validated. Tests on four elastomeric materials and seven lubricating greases have been achieved. Post irradiation examination can successfully determine the evolution of the most relevant mechanical parameters as a function of the absorbed dose in specific irradiation conditions. As a consequence, products performance can be comparatively evaluated [13].

The definition of radiation resistance is a delicate and challenging topic to be developed. First of all, it is necessary to define dose thresholds that are correlated to significant variations of relevant mechanical properties. Dose thresholds depend indeed on the chosen thresholds of material damage, referred to as "end-point". End-point definitions are very important to establish the stability of a component performance in application. Nevertheless, they are scarcely defined in the literature. In the present study they are currently under examination. Elongation at break value equal or below 100% for elastomers and consistency NLGI class variation for greases can be considered as preliminary end-of-life conditions for radiation stability considerations and materials application in the projects. More refined investigations dedicated to specific applications of these materials are ongoing.

The important role of several irradiation parameters in determining radiation-induced damages is confirmed by the presented results.

Comparing the present radiation hardness data with the data found in the producer's data sheets, which refer to traditional gamma sources, it can be observed that irradiation in the reactor, given the same amount of total dose, appears to be more effective in damaging some of the selected materials. This evidence largely support the need for producing data achieved in mixed neutron and gamma fields, being different from the normally available ones usually obtained using gamma fields only. Moreover, parameters like the typology and energy of radiation particle, the dose rate, the oxygen conditions, and the temperature should be varied and investigated for a better understanding of the radiation-damage mechanisms and effects. New protocols for O-rings and greases irradiation are being developed to evaluate the damage dependence on several irradiation parameters, adding important information to the present study. Radiation resistance definitions commonly used consider only the total amount of absorbed dose as the significant parameter. The concept of radiation resistance itself should be reformulated in the light of the present data and should include a specification of the employed irradiation conditions as well.

The results of the present work, in addition to the scientific significance, provide a useful input for the ESS and the SPES project. These facilities, being under construction, require the solution of non-metallic materials challenges which are more severe than those previously encountered. New irradiation and testing protocols will be developed to investigate the radiation resistance in reactor mixed fields of different categories of materials as well, for example cable insulators.

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