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Experimental study of consistency degradation of different greases in mixed neutron and gamma radiation

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ABSTRACT

Many of the moving components in accelerator and target environments require lubrication. Lubricants in such environments are exposed to high fluxes of secondary radiation, which originates from beam interactions with the target and from beam losses. The secondary radiation is a mix of components, which can include significant fractions of neutrons. Lubricants are radiation-sensitive polymeric materials. The radiation-induced modifications of their structure reduce their service lifetime and impose additional facility maintenance, which is complicated by the environmental radioactivity. The study of the lubricants radiation resistance is therefore necessary for the construction of new generation accelerators and target systems. Nevertheless, data collected in mixed radiation fields are scarce. Nine commercial greases were irradiated at a TRIGA Mark II Research Reactor to serve for the construction of new accelerator projects like the European Spallation Source (ESS) at Lund (Sweden) and Selective Production of Exotic Species (SPES) at Legnaro, (Italy). Mixed neutron and gamma doses ranging from 0.1 MGy to 9.0 MGy were delivered to the greases. For an experimental quantification of their degradation, consistency was measured. Two of the greases remained stable, while the others became fluid. Post-irradiation examinations evidence the cleavage of the polymeric structure as the dominant radiation effect. Dose and fluence limits for the use of each product are presented. Apart from the scientific significance, the results represent an original and useful reference in selecting radiation resistant greases for accelerator and target applications.

1. Introduction

Polymeric materials used in high power accelerator and target environments are exposed to mixed radiation fields. Neutrons and gamma can represent significant components of these fields. The absorbed dose due to different radiations causes structural modifications of these materials. Radiations affect polymers mainly through the basic mechanisms of cleavage and cross-linking of macromolecular chains [1, 2, 3]. Therefore, significant radiation-induced modifications of their physical and mechanical properties can be induced, which could lead to a premature failure of the components.

The European Spallation Source (ESS) project is under construction in Lund, Sweden, aiming at becoming the brightest neutron source in the world [4]. The Selective Production of Exotic Species (SPES) facility is being built in Legnaro, Italy aiming at producing intense Radioactive Ion Beams via fission reactions occurring in its target [5, 6]. Both of their targets will produce intense mixed fields of neutrons and gamma radiation. Elastomeric O-rings, lubricating oils and greases are necessarily employed in their target areas. As they are exposed to secondary neutrons and gammas, their modifications must be evaluated for safe and reliable facility operations. Unfortunately, radiation damage data on polymers in neutron fields and in mixed radiation fields are scarce in the literature.

Most of the available data originate from technical reports produced by scientific organisations and by companies manufacturing products certified to be radiation-resistant. The Yellow Reports from CERN are one of the widely used references concerning the radiation resistance of polymeric materials. Results on seven types of elastomeric O-rings

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available on the CERN store shelves and two types of lubricating oils irradiated up to several MGy of gamma dose are reported in Ref. [7]. Greases were not analysed.

In an ITER report [8], two commercial elastomeric gaskets and four greases declared by the producer as radiation-hard were tested up to high gamma doses from 10 MGy to 100 MGy. A recent work [9] identifies O-rings, lubricants and cable insulators among the most critical components in the ITER design. Radiation effects on oils and greases evaluated in the viewpoint of their chemical composition are reported in Refs. [1] and [10]. Neutron and gamma irradiation data on twelve solid lubricants are reported in [11].

The available literature on the radiation effects in polymers is not satisfactory for the construction of new accelerators and target facilities. In fact, most of the studies were performed more than a decade ago. Considering the rapid evolution of the industrial product development, most of the tested products are now outdated. Furthermore, most of the literature and data available by manufacturers lack details about the irradiation conditions such as radiation type, dose rate, presence of oxygen and linear energy transfer (LET), which influence radiation effects in polymers [12, 13]. Lastly, most of the studies used gamma sources, although accelerator and target environments generate mixed radiation fields. In some of them neutrons represent the dominant component.

Gamma sources have been preferred to neutron sources for materials testing because of their wider availability and because the irradiated samples maintain a much lower residual activation. It has been believed that the radiation-induced damage depends on the total amount of absorbed dose only, irrespectively of the type of radiation [7, 14, 15]. Accordingly, the polymers radiation resistance has been expressed in terms of gamma dose and directly translated to neutron and mixed field dose. Nevertheless, the equivalence of gamma and neutron doses has not been theoretically verified nor experimentally validated.

Contrary to the assumed equivalence between gamma and neutron doses, it is reported in Ref. [1] that the effects of mixed reactor radiation in polymers might differ from that of gamma alone. The data here presented challenge this long believed equivalence as well. In a UKAEA report [16], the need for establishing more precisely the relative effects of similar doses of neutrons and gammas is addressed. In a seminal study performed at ORNL, the radiation damage induced in elastomers by reactor radiation is compared with the damage produced by different gamma sources. It was reported that similar damages are induced by doses of the two radiations that can differ of a factor two [17]. In a more recent work, the understanding of radiation effects in polymers and their dependence on the radiation typology are clearly stated as one of the most important problems to be solved [18]. According to Rivaton and Arnold, fast neutrons effects on organic materials are not fully understood despite their relevance in the nuclear engineering and new investigations are needed [19]. The full comprehension of the effects of intense neutron fields in polymers is reported as a requirement for their application in nuclear technology by Bonin et al. [20]. The need for research on fast neutron effects on polymers is reported by Seguchi et al. [3]. They report as well the lack of such studies in the literature, which is dominated by gamma radiation studies.

For the mentioned reasons, radiation damage on polymeric materials has to be further investigated. In particular, mixed radiation data are necessary for the design and construction of new facilities producing intense neutron fields. We dedicated a previous study to the development of a methodology for testing elastomeric O-rings irradiated in neutron and gamma mixed fields [21]. A following study was dedicated to the experimental determination of the end-of-life conditions of a specific EPDM O-ring to be installed in a gate valve of the SPES facility. It represents an example of the design of a component which operates in a mixed neutron and gamma radiation environment [22]. The management of the SPES facility as an intense neutron source is detailed in [23]. The problem of the radiation resistance of lubricants is briefly mentioned therein.

In this framework, the present study is dedicated to lubricating greases. A methodology for greases irradiation in a mixed neutron and gamma reactor field was developed. Post-irradiation examinations based on consistency evaluations were completed. The chosen definition of dose thresholds allowed the selected products to be comparatively evaluated.

In Section 2.1 a description of the commercial products selected for this study can be found as well as their most relevant properties. Seven of the most important grease producers in the world have been considered. In Section 2.2 a description of the mixed neutron and gamma irradiation facility of a TRIGA Mark II nuclear research reactor used for sample irradiation is reported. In Section 2.3 a description of the dose components absorbed by the different greases in the neutron and gamma irradiation mixed field is detailed. The methodology developed to irradiate and test the grease evolution with dose is reported in Section 2.4. Consistency is chosen as the most significant indicator of radiation-induced grease deterioration, as reported in Section 2.5. Results on nine greases irradiated in a range between 0.1 MGy and 9 MGy of absorbed dose are shown in Section 3. Experimental evidences of radiation-induced cleavage of the grease structure are reported in Section 4.1 whereas considerations on the grease radiation resistance as a function of their chemical compositions are reported in Section 4.2. In the discussion section, the greases are compared based on their consistency. Dose and particle fluence thresholds are defined to estimate the usability of each grease, as reported in Section 4.3. Consistency results are compared with the radiation resistance declarations of the producers in Section 4.4. The possibility of using the present results to foresee the end-of-life grease conditions in specific applications is discussed in Section 4.5. Conclusions are drawn in Section 5.

2. Product selection and testing methodology

Greases are semi-fluid to solid systems originating from the dispersion of a thickener in a liquid oil [1]. A standard grease contains about 85% base oil, 10% thickener and 5% other additives. Greases are complex multi-phase systems whose chemical and physical properties originate from the coexistence and interaction of their components [24].

The radiation resistance of base oils is reported to predominantly depend on their aromatic content. A higher content of aromatic structures is associated to a higher stability of the oil viscosity as a function of the absorbed dose. For this reason, greases based on aromatic and polyether oils are considered more radiation resistant than mineral oil based ones [1] [10]. However, the experimental data here presented challenge this general statement.

The academic literature on the radiation resistance of lubricating greases is very limited, as reported by R.M. Mortier et al. [24]. For this reason, in the present paper all the available information provided by the producers is used as reference too. Radiation effects in greases are complicated by the interaction of the base oil with the thickener and the additives and the overall effect is expected to depend not only on the effects on its bulk components, but on their complex interaction as well.

Since the available information on the grease radiation resistance is so limited, a wide and qualified spectrum of products to be tested was necessary. A market research has been completed to select: i) experienced companies certifying a quality control on the grease production; ii) greases differing in the chemical compositions of their components; iii) both greases declared as radiation resistant by the producer and generic ones; iv) greases having different features: vacuum compatibility, extreme pressure resistance and extreme duration.

These criteria lead to a selection of high-quality greases suitable for different applications in accelerator and target environments.

2.1. Selected lubricating greases

Nine commercial greases were selected from the market (see Table 1). Indications about their chemical composition are provided by the producer and reported in data sheets. Consistency, whose value is reported as a NLGI class, is one of the most relevant properties characterizing the grease [24].

The nine selected greases and their major properties are listed below:

- AFB-LF is produced by THK (Japan). It is a general-purpose grease for bearing applications, manufactured using a refined mineral oil and a Li based thickener. It features extreme pressure resistance and high mechanical stability.
- Apiezon M is produced by M&I Materials (United Kingdom).[1] It is developed for vacuum use and it is declared as radiation resistant up to about 1 MGy of dose delivered using a 4 MeV electron beam. Apiezon M is manufactured with a hydrocarbon oil and it is halogen free. This is an advantage for applications in radiation environment, because halogens can evolve acids. Apiezon M is manufactured without a thickener. For this reason, it is more properly referred as a very viscous fluid rather than a grease.
- FAG Arcanol LOAD 220 is produced by Schaeffler (Germany). It is developed for ball and roller bearings. It is manufactured using a mineral oil and a mixed thickener. It features high load resistance and declared durable performance.
- Grizzlygrease No. 1 is produced by Lubricant Consultant GmbH Lubcon (Germany). It is manufactured using a mineral oil and a Li/Ca special soap. It is developed for gears application and declared as resistant to gamma radiation up to a dose level of 1.2 MGy.
- Klüberlub BE 41-542 is produced by Klüber Lubrication (Germany). It is manufactured using a mineral oil and a special lithium soap. It is defined as heavy-duty product, developed for high-load rolling bearings and featuring extreme pressure (EP) resistance.
- Krytox 240 AC is produced by Chemours Company (USA).[2] Krytox fluoropolymers are high-performance lubricants initially developed for the aerospace industry. Now they have a broader range of applications, from automotive to electronics. Krytox is a perfluoropolyether (PFPE) whose polymer chain is completely saturated with fluorine. According to the producer declarations, it contains carbon, oxygen and fluorine only. Krytox 240 AC grease is compounded using polytetrafluoroethylene (PTFE) as thickener. It is selected in the present work in view of its claimed outstanding performance and of its hydrogen-free composition. The grease and its base oil were irradiated using a ^{60}Co gamma source and then tested by the producer. A progressive grease softening is reported as a function of the absorbed dose. Reported consistency variations are generally not exceeding a 10%-15% compared to the unirradiated samples. The radiation stability of the base oil was evaluated in a nuclear reactor, up to a maximum value of 1 MGy.
- Petamo GHY 133 N is produced by Klüber Lubrication. It is manufactured using a mineral oil and a polyurea thickener. It is declared as a long-term and high-temperature grease for rolling bearings.
- RG-42R-1 is produced by MORESCO Corporation (Japan). It is declared as radiation resistant up to high gamma doses. Tests are performed by the producer on its base oil only. It is considered as representative of the whole grease in a simplistic way. Absorbed dose levels up to 30 MGy are delivered to the oil using a ^{60}Co gamma source with a dose rate of 0.01 MGy/h. The viscosity evolution of the oil as a function of the dose is evaluated. A dose threshold of 15 MGy is determined accordingly. The radiation resistance of the grease is supposed to be equal to the radiation resistance of the oil by the producer. Most of the MORESCO radiation resistant products including RG-42R-1 are manufactured using a polyphenylether base oil and a polycarbonate thickener.
- Turmopolgrease 2 produced by Lubricant Consultant GmbH Lubcon is a grease manufactured with a polyglicol oil and a lithium based soap. It features a very long lubrication service compared to conventional products with the same composition.

Table 1
The lubricating greases selected from the market for irradiation and testing are listed. The composition of the base oil and of the thickener is specified. Consistency expressed in NLGI grade is reported. Radiation resistance, when declared by the producer, is reported. The information originates from the data sheets of the products provided by the suppliers.

Producer and product	Base oil	Thick.	Cons. NLGI	Rad-hardness
AFB-LF	Mineral	Li	2	Not declared
Apiezon M	Hydrocarbon	—	—	1 MGy, electr.
FAG Arcanol LOAD 220	Mineral	Mixed	1-2	Not declared
Grizzlygrease No.1	Mineral	Li/Ca	0	1.2 MGy, γ
Klüberlub BE 41-542	Mineral	Li	2	Not declared
Krytox 240 AC	PFPE	PTFE	2	1 MGy, reactor
RG-42R-1	PPE	PC	1	15 MGy, γ
Petamo GHY 133 N	Mineral	Polyurea	2	Not declared
Turmopolgrease 2	Polyglicol	Li	2	Not declared

Table 2
Elemental composition of the selected greases in weight percent is obtained with CHN analyses. The percentage of the mass not measured by the CHN analysis is reported as well. The measurement errors are contained in the second decimal figure of the quoted results.

Product	C %	H %	N %	Others %
AFB-LF	82.75	14.13	0.00	3.12
Apiezon M	86.19	13.70	0.11	0.00
FAG Arcanol LOAD 220	80.57	13.15	0.25	6.03
Grizzlygrease No.1	79.23	11.63	0.18	8.96
Klüberlub BE 41-542	82.50	12.29	0.58	4.63
Krytox 240 AC	21.03	0.00	1.01	77.96
Petamo GHY 133 N	84.26	12.42	1.23	2.09
RG-42R-1	78.50	9.67	0.00	11.83
Turmopolgrease 2	62.97	10.71	0.00	26.32

The number of greases declared as radiation resistance on the world market is very limited.

As described in details in Paragraph 2.2, the dose absorbed by greases in a fast neutron field highly depends on its light element composition. In particular, it is proportional to the hydrogen content. For this reason, the mass fraction of some relevant light elements composing the greases is measured by CHN chemical analysis. The percent content of carbon, hydrogen and nitrogen in each product is measured and reported in Table 2.

The hydrogen concentration for the selected products ranges from 0.00% to 14.13%, depending on their chemical composition. Krytox 240 AC is hydrogen-free, as declared by the producer. It is completely saturated with fluorine, whose amount is about 70% of the total mass.

The hydrogen mass percentage of MORESCO RG-42R-1 product is 9.67%. It is compatible with the chemical composition of its oil, which is polyphenylether based. The hydrogen mass concentration of mineral oil based products ranges from 11.63% and 14.22%. The results of these analyses are used to simulate the composition of the materials for dosimetry calculations

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[1] Apiezon®is a registered trademark.

[2] Chemours™ and Krytox™ are trademarks.

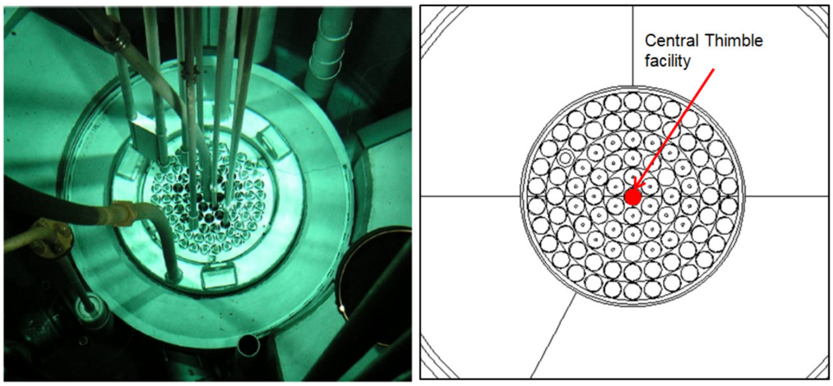

Fig. 1. The in-core Central Thimble irradiation facility of the TRIGA Mark II nuclear reactor of the University of Pavia. Left: a picture of the reactor core. The aluminum pipe of the Central Thimble, reaching the middle of the reactor core, can be seen. Right: a horizontal cross-section of the reactor core as modelled with the simulation program MCNP5. The Central Thimble facility is indicated.

Table 3
Irradiation conditions of the Central Thimble facility of the TRIGA Mark II nuclear reactor of the University of Pavia. Parameters refer to the nominal working power of 250 kW. Fluxes are calculated using a reactor model realized with the simulation Monte Carlo code MCNP5. The fast neutron flux refers to neutrons having energy higher than 0.5 MeV. The gamma flux considers the fission prompt gammas and the photons from radiative capture of neutrons on the reactor materials. Since the gammas produced by the fission product decay are not simulated by MCNP5, the gamma flux is underestimated.

Parameter	Value
Neutron flux	1.72 10^{13} neutron/(cm^2 s)
Fast neutron flux	3.80 10^{12} neutron/(cm^2 s)
Gamma flux	1.65 10^{13} photon/(cm^2 s)
Temperature range	50 °C – 70 °C
Atmosphere	Air at atmospheric pressure

2.2. *Irradiation conditions, fluxes and spectra*

The TRIGA Mark II nuclear reactor of the University of Pavia, Italy is equipped with irradiation facilities for research. The Central Thimble (see Fig. 1) is an in-core facility reaching the centre of the fuel elements.

Radiation components in the Central Thimble originate from several reactions occurring in the reactor core. Nuclear fission is the main source of neutrons and photons. Most of the gammas are produced by fission reactions and by the decay of radioactive fission products. They are referred to as prompt gammas and delayed gammas respectively. In addition, a minor gamma component is due to radiative capture reactions in the materials constituting the reactor [25].

In the present work, radiation fields and dose components are calculated with a simulation of the reactor [26] realized with the Monte Carlo code MCNP5 [27]. In a previous experimental campaign, the Central Thimble neutron spectrum was measured. The calculated neutron spectrum agrees with the measured one [28]. Accordingly, the calculated neutron spectrum can be considered a reliable representation of the measured one. On the contrary, the calculated gamma flux is underestimated because MCNP5 does not account for the fission products decay. Based on existing measurements on similar TRIGA Mark II reactors, the missing gamma component can be estimated. It can represent up to 30% of the total gamma component [29]. The systematic error on the gamma dose is evaluated accordingly. Table 3 summarizes the Central Thimble calculated parameters.

Fig. 2 shows the calculated neutron energy spectrum in the Central Thimble. The spectrum is normalized to the nominal reactor working power of 250 kW, which is associated to a source intensity of $1.9 \cdot 10^{16}$ n/s. Two main components are present. The fast component refers here to neutrons with energy higher than 0.5 MeV. Fast neutrons originate from fission reactions. Their distribution in the Central Thimble facility is comparable to a typical fission spectrum. An important component of slower neutrons in the thermal and epithermal energy range is present as well. They originate from the moderation of fast neutrons in the reactor moderator and structures. The neutron dose absorbed by the irradiated greases depends on the specific neutron spectrum.

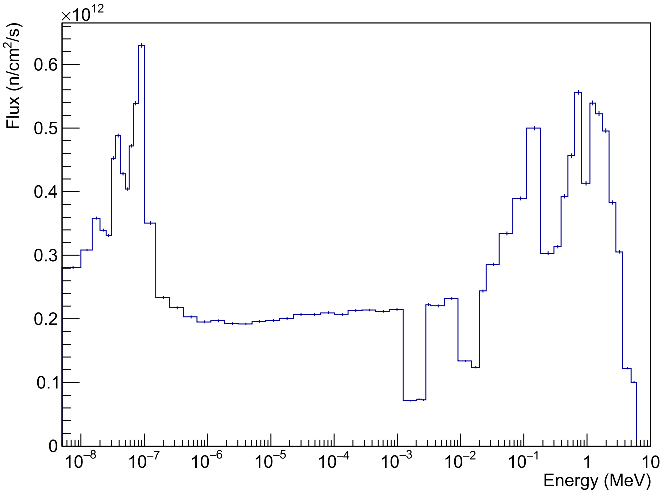

Fig. 2. The neutron energy spectrum in the Central Thimble facility calculated with MCNP5. The spectrum is normalized to the nominal reactor power of 250 kW. The fast neutron component and the thermal-epithermal one are dominant.

2.3. *Dosimetry calculations*

The neutron and photon dose components absorbed by each irradiated grease are calculated using MCNP5. The grease composition in the simulation is based on the CHN analyses results (see Table 2). Additional assumptions are made to model the missing composition fraction. For Krytox 240 AC, which is a fluorinated material, the missing mass is assumed to be fluorine (70.4%) and oxygen (7.56%). These assumptions are based on the base oil and thickener chemical compositions, as declared by the producer. For all the other greases, the remaining mass is assumed to be oxygen. Metallic traces are present in the products in most cases. However, their relevance in the overall dosimetry calculation is negligible. As a first approximation, metallic traces are neglected. The systematic error introduced by these hypotheses in the dosimetry calculations is estimated by changing the assumed compositions in different ways. However, since the neutron dose is mostly related to the hydrogen content, whose amount is measured, the error due to the assumptions on the composition is not larger than few percent.

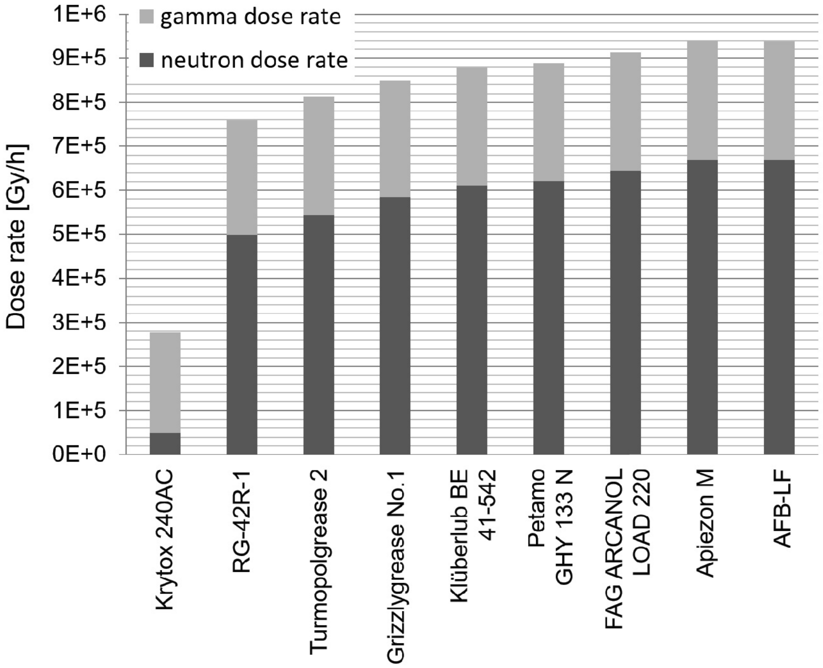

Fig. 3. Dose rates calculated with MCNP5 for greases irradiated in the Central Thimble facility at the nominal working power of 250 kW. Greases are ordered from left to right according to the increasing hydrogen content, ranging from 0.00% (Krytox 240 AC) to 14.13% (AFB-LF). The neutron dose rate component is in dark grey, the gamma one in pale grey. The overall systematic error is estimated to be lower than 15%. The statistical error on the simulated results is negligible compared to the systematic one.

The dose absorbed in a neutron and gamma mixed field highly depends on the composition of the irradiated materials and on the radiation energy spectrum. For this reason, the dosimetry considerations here reported specifically refer to the selected greases irradiated in the Central Thimble. The calculated total dose rates for the greases are reported in Fig. 3. The dose rate is separated in its two main neutron and gamma components.

Except for Krytox 240 AC, whose hydrogen content is negligible, for the other greases the total dose rate ranges from 0.76 MGy/h to 0.94 MGy/h. The difference depends on the neutron contribution, ranging from 0.50 MGy/h to 0.67 MGy/h. It is proportional to the hydrogen mass content, ranging from 9.67% to 14.13%. The neutron components dominate, representing 65%-71% of the total. The gamma component is about 0.26 MGy/h for all the greases. It is roughly irrespective of the specific material composition.

Because of its hydrogen-free composition, Krytox 240 AC behave differently in this neutron field. Its total dose rate is 0.28 MGy/h, about three times lower compared to the other greases. This is due to the neutron component, which is more than one order of magnitude lower. The gamma component dominates, representing about 93% of the total. Products containing halogens are usually not employed in radiation environments because they can produce acids during irradiation. Nevertheless, fluorinated products are interesting for applications in neutron fields in view of their low hydrogen content. This is the reason why a fluorinated product has been selected for the present study.

The neutron dose component is proportional to the hydrogen content for all the greases. This is because the main energy release mechanism is the elastic scattering of fast neutron on light nuclei contained in the grease. The average energy transferred to a nucleus in this process increases when the mass of the target nucleus decreases. For this reason, the process is mostly effective on hydrogen. Moreover, the average energy transferred via elastic scattering is proportional to the incoming neutron energy. For this reason, the average energy transferred by fast neutrons is several orders of magnitude higher than the one transferred by thermal neutrons by the same mechanism[30]. High-energy secondary protons originating from neutron scattering on hydrogen deliver a large fraction of the total dose.

The systematic error on the total absorbed dose is estimated to be lower than 15%. It depends on the agreement between the measurements and the simulated model. However, the total absorbed dose is underestimated because of the fraction of the gamma component that is not accounted for by the code. The statistical error on the simulated results is negligible compared to the systematic one.

In the present work, the total amount of absorbed dose is chosen as the parameter to which the grease consistency evolution is referred. In addition, radiation effects in greases are referred as well to the total neutron and gamma particle fluence to which the samples are exposed during irradiation. Table 4 shows the dose values absorbed by the irradiated greases and the total neutron and gamma fluence. Those values depend on the particle fluxes in the irradiation facility, as reported in Table 3 and on the exposure time only, irrespective of the grease composition. In fact the irradiated grease samples do not significantly perturb the particle fluxes in the facility because of their small size.

2.4. *Experimental methodology*

The nine greases were irradiated for exposure times ranging between 10 minutes and 10 hours. The most stable greases were selected for 5 h and 10 h long irradiations. The irradiations are realized in the Central Thimble facility at the nominal reactor power of 250 kW. Table 4 reports the chosen irradiation times and the corresponding absorbed doses. Absorbed doses range from about 0.1 MGy to about 9 MGy. The samples are irradiated in air at atmospheric pressure.

A single irradiation was completed on Krytox 240 AC grease. An irradiation time of 3 h and 30 minutes was selected in view of its fluorinated composition. Additional irradiations were not possible because of the limited amount of available product, whose commercial price is extremely high compared to the other ones.

Qualitative preliminary tests were performed on irradiated greases to design a safe an efficient set-up. One test evaluates the radiation-induced grease mobility. Small plastic containers were filled with dif-

Table 4

Doses absorbed by the selected greases in MGy. Corresponding irradiation times in the Central Thimble facility of the TRIGA Mark II reactor are reported. Krytox 240 AC is not reported here. Since its dose rate in the facility is much lower compared to the other ones, a single irradiation time of 3 hours and 30 minutes was chosen, corresponding to a total dose of 0.99 MGy. Total neutron and gamma fluence to which the samples are exposed is reported as well as a function of the irradiation time.

Irradiation time	10 min	30 min	1 h	2 h	5 h	10 h
Product						
AFB-LF (MGy)	—	0.47	0.94	1.88	—	—
Apiezon M (MGy)	0.16	0.47	0.94	1.88	4.70	—
FAG Arcanol LOAD 220 (MGy)	0.15	0.46	0.91	1.83	—	—
Grizzlygrease No.1 (MGy)	0.14	0.42	0.85	1.70	—	—
Klüberlub BE 41-542 (MGy)	0.15	0.44	0.88	1.75	4.39	—
Petamo GHY 133 N (MGy)	0.15	0.44	0.89	1.78	4.44	8.89
RG-42R-1 (MGy)	—	0.38	0.76	1.52	3.80	7.60
Turmopolgrease 2 (MGy)	0.13	0.41	0.81	1.63	—	—
Neutron fluence (10^{16} par cm^{-2})	1.03	3.1	6.2	12.4	31.0	62.0
Gamma fluence (10^{16} par cm^{-2})	0.99	3.0	5.9	11.9	29.7	59.4

Fig. 4. Left: a syringe filled with grease, broken due to the radiation-induced gas pressure. Right: a section view of the syringe after irradiation. The radiation-induced grease mobility can be observed. The scattering of the grease inside the syringe volume is due to the production of gas bubbles.

ferent amounts of grease ranging from 20% to 80% of the total volume. An important radiation-induced gas evolution is observed, promoting a high grease mobility in the containers (see Fig. 4). The developed pressure due to the gas production in the set-up can reach critical levels, sometimes damaging and breaking the container.

Radioactive nuclides are produced in the irradiated greases due to neutron capture reactions. Because of their residual activation, irradiated greases must be handled by qualified operators according to the existing radiation protection regulation. Seven days after irradiation the contact dose rate ranges between 0.1 μSv/h and 0.3 μSv/h for all the greases. This is comparable with the natural radiation background, therefore the grease samples can be safely tested after irradiation.

Based on the preliminary tests, a 20 mL disposable plastic syringe is chosen as irradiation set-up. A maximum of 10 mL of grease is irradiated, to avoid critical gas evolution and excessive mobility. The grease is distributed as a layer on the inner syringe surface. The syringe is placed in a cylindrical aluminum container routinely used for sample irradiation. Fig. 5 shows the set-up.

2.5. Consistency tests

Consistency is widely considered as the most important property of a lubricating grease and marks the difference between liquid oils and greases [24]. The presence of the thickener gives the grease a solid character and a rigidity commonly referred to as consistency. Consistency is measured for both irradiated and unirradiated grease samples according to ASTM D217-02 and ASTM D1403-02 standards. Consistency is therein defined as *the degree of resistance to movement under stress*. It is measured with a penetrometer equipped with a cone whose tip penetrates the flat grease surface (see Fig. 6). The cone is free to fall into the grease under gravity for 5 seconds. Consistency measures the cone penetration in tenths of millimeters. A semi-automatic precision penetrometer by S.D.M. Apparecchi Scientifici s.r.l. equipped with a one-quarter scale cone is used in this work. The one-quarter scale equipment as described in ASTM D1403 allows a consistency test to be completed on a sample of about 5 mL only. This is an advantage considering the limited available space in the irradiation facility and the need to reduce as much as possible the amount of produced radioactive waste.

Fig. 5. The set-up used to irradiate grease samples in the Central Thimble facility. A disposable plastic 20 mL syringe is filled with about 10 mL of grease. The syringe is placed in an aluminum container.

According to the standard, the grease must be worked using a manipulator before being tested (see Fig. 7). The grease is subjected to 60 full double strokes in one minute. This manipulation is necessary to perform a worked penetration test. The consistency measured after manipulation is referred to as worked consistency.

The National Lubricating Grease Institute (NLGI) classifies greases based on their worked consistency. NLGI grades are universally accepted and used by producers and final users. NLGI grades range from 000 to 6. Grade 000 indicates a soft and almost fluid material condition, corresponding to high penetrations (445-475 mm/10). Grade 6 indicates a hard solid-like material condition, corresponding to low penetrations (85-115 mm/10). For the greases selected in the present study, a 10% consistency variation approximately corresponds to a variation of one grade.

Fig. 6. The semi-automatic penetrometer by S.D.M. Srl used for consistency measurements. Right: the penetration cone positioned over the flat surface of the grease, before the measurement. The one-quarter scale equipment is used.

Fig. 7. The one-quarter scale equipment used to manipulate the grease before the measurement of consistency. The grease is subjected to 60 full double strokes in about one minute.

The one-quarter scale equipment used in this work can measure NLGI grades ranging from 0 to 4. As reported. Measured values lower than 0 in NLGI class will be here referred to as out-of-scale. Measured values higher than 4 were not observed.

3. Results

Consistency variations of the irradiated samples relative to the unirradiated ones are reported as a function of the total absorbed dose (see Fig. 8). The relative consistency values are calculated as follows:

$$C_{rel}(D) = \frac{C(D)}{C_0} \tag{1}$$

C(D) is the consistency value (in mm/10) of the grease irradiated at a total dose D. C_0 is the consistency value (in mm/10) of the unirradiated grease. The error associated to $C_{rel}(D)$ is estimated to be lower than 5%, basing on repeated measurements on unirradiated samples.

Six out of the nine tested greases experience severe radiation-induced consistency increase as a function of the dose. They become almost fluid between 0.4 MGy and 5 MGy. The condition and appearance of these grease samples after irradiation are completely different from the unirradiated ones. The consistency increase is remarkable, exceeding the range of the instrument. These exceeding values are anyway used in Equation 1 to estimate a relative consistency variation. They are associated to a fluid grease state and they are in the present paper referred to as out-of-scale values. They are marked by dashed lines in Fig. 8.

Post-irradiation consistency results can be analysed with reference to their percentage deviation from their unirradiated samples consistency, recalling that a 10% consistency variation is approximately associated to a change in NLGI grade.

Three groups of products can be distinguished according to their consistency modification with absorbed dose:

1. Huge consistency increases are reported for AFB-LF, Grizzlygrease No.1, Krytox 240 AC and Turmopolgrease 2 at dose values lower than 1 MGy.

   AFB-LF consistency increases of 25% at 0.94 MGy. It becomes fluid at 1.9 MGy, showing a 46% increase in consistency.

   Grizzlygrease No.1 consistency increases of 25% at 0.4 MGy. This value is out-of-scale and the grease becomes fluid (see Fig. 9). The grease remains steadily fluid up to about 1.7 MGy.

   Krytox 240 AC becomes fluid at 0.97 MGy, showing a 45% consistency increase. Acid gases evolved during irradiation due to the fluorine content led to the corrosion of the aluminum container (see Fig. 10).

   Turmopolgrease 2 consistency increases of 37% at 0.8 MGy. It becomes fluid at 1.6 MGy of dose, showing a 60% consistency increase.

2. Apiezon M, FAG Arcanol LOAD 220 and Klüberlub BE 41-542 remain rather stable up to 1.0 MGy. Their consistency remarkably increases between 1.5 MGy and 5 MGy.

   FAG Arcanol LOAD 220 is stable up to 0.9 MGy. It abruptly becomes fluid at 1.8 MGy, showing a 40% consistency increase.

   Klüberlub BE 41-542 exhibits a progressive consistency increase as a function of the dose. Consistency value increases of 7% at 0.9 MGy and of 10% at 1.8 MGy of dose. It becomes fluid at 4.4 MGy, showing a 30% consistency increase.

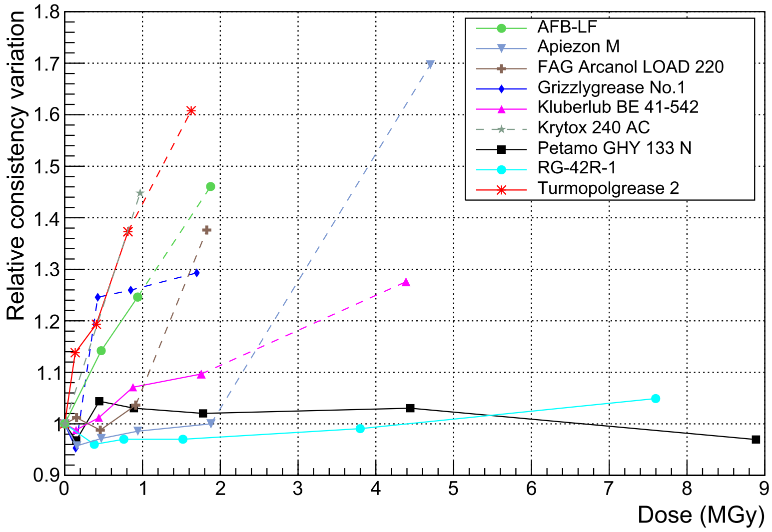

Fig. 8. Relative consistency value $C_{rel}(D)$ as a function of the total absorbed dose for the nine selected greases. Dashed lines indicate consistency values exceeding the maximum instrument range, corresponding to an almost fluid material state. High consistency values are associated to softer greases, which correspond to lower NLGI consistency grades.

Fig. 9. Post-irradiation consistency test of Grizzlygrease No.1. Top left: grease irradiated at 0.15 MGy. Consistency is comparable to the unirradiated one. Bottom left: Grizzlygrease No.1 irradiated at 0.45 MGy. A remarkable consistency increase can be observed. The measured consistency value is out of the instrument scale. The irradiated grease becomes fluid. Right: the grease irradiated at 0.45 MGy after penetration measurement. The grease drips from the penetrometer tip.

Apiezon M does not exhibit significant consistency variations up to 1.9 MGy. It abruptly becomes fluid at 4.7 MGy, showing a 70% consistency increase.

3. **Petamo GHY 133 N** and **RG-42R-1** do not exhibit significant consistency variations up to the maximum investigated dose values of 8.9 MGy and 7.6 MGy respectively.

The colour of some greases is darkened by irradiation. It is particularly evident for Petamo GHY 133 N (see Fig. 11), whose colour turns from yellowish (unirradiated sample) to black at about 0.4 MGy.

Fig. 10. Left: Krytox 240 AC sample irradiated at 0.97 MGy (3 h and 30 min). Right: the aluminum set-up used for irradiation. The stopper has been corroded by the acid gases evolved.

4. Discussion

4.1. Cleavage observation in the grease structure

Greases are complex multi-phase systems. For this reason, the consistency evolution with dose is expected to be complex too. Radiation effects in greases result from the interaction of several damage mechanisms. Two main effects characterize the interaction of radiation with polymers at the structural level: cleavage and cross-linking of the polymeric chains. As reported by R.O. Bolt and J.G. Carrol [1], the first radiation effect expected in greases is the damage of the gelling structure of the thickener, causing a consistency increase. Radiation can induce fractures in the soap fibres, which become unable to maintain a gelling structure. At higher dose levels, a consistency decrease is expected because of the increased viscosity of its base oil due to polymeric chains cross-linking. The former behaviour was observed for most of the investigated products, whereas the latter was not observed in the considered irradiation conditions.

According to the literature, the presence of aromatic groups in the base oil should determine the radiation-resistance of a grease. However, the consistency results don't allow the grease chemical structure and its radiation resistance to be easily correlated. The collected data confirm the complexity of the radiation interaction with multi-phase systems.

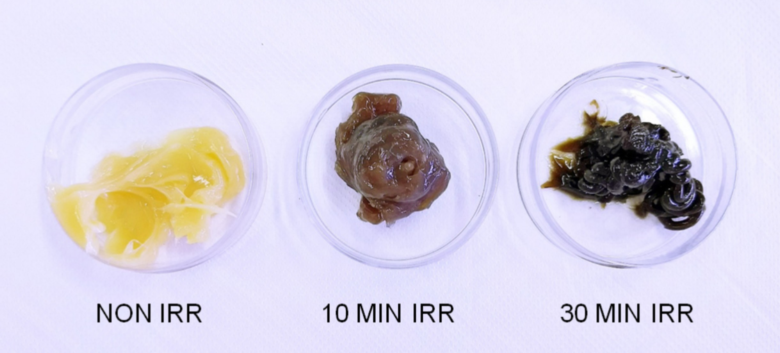

Fig. 11. Petamo GHY 133 N grease samples. From left to right: an unirradiated sample, a sample irradiated at 0.15 MGy (10 min irradiation) and a sample irradiated at 0.44 MGy (30 min irradiation). A progressive grease darkening can be observed, from yellowish to completely black. Samples irradiated at higher dose levels have the same colour of the one irradiated at 0.44 MGy.

The tested greases feature either a stable consistency or a consistency increase with dose. Seven out of the nine irradiated greases become fluid and drip from the testing instrument under gravity (See Fig. 9). By definition, a grease should remain in place as a solid body under gravity [24]. Grease fluidization is an indicator of severe material damage, probably related to a complete damage of the thickener structure. The thickener is a radiation-sensitive component and for this reason it is relevant in determining the overall radiation-resistance of the grease.

Apiezon M behaviour is discussed separately because it is manufactured without a thickener. For this reason, its fluidization at 4.7 MGy is not related to the thickener disruption and might be related to the oil structure cleavage.

The qualitative preliminary tests provide indirect information on the grease radiation effects too. Grease mobility is highly promoted by irradiation for all the tested products. The intense gas evolution could originate from the cleavage of the polymeric chains. The gas evolved during irradiation leads to the formation of bubbles, which modify the distribution of the grease in the set-up. Acid gases are evolved by Krytox 240 AC because of its fluorinated composition.

In conclusion, cleavage dominates as damage mechanism for the greases here analyzed. Further understanding of the mechanisms of radiation interaction at the structural scale is not possible based on consistency results only. Greases are complicated systems, whose chemical and physical characteristics cannot be easily modelled. The lack of academic base research into the grease fundamentals evidences the need for further investigation [24].

4.2. Correlation between radiation effects and chemical composition

According to the literature, the grease radiation stability should be predominantly determined by its base oil chemical composition. For example, polyphenylether based products are reported to be more radiation-resistant than mineral oil based ones [1, 10]. The outstanding stability of RG-42R-1 complies with this consideration. However, there are reasons to believe that radiation stability depends on a more complex interaction between the chemical nature of the base oil, the thickener and the additives. This is supported by some of the results here reported.

Five of the tested greases (AFB-LF, FAG Arcanol LOAD 220, Grizzlygrease No.1, Klüberlub BE 41-542 and Petamo GHY 133 N) are manufactured with mineral oils but different thickeners. Despite the similar composition of their base oil, they show very different behaviour, maybe thanks to the presence of different thickeners. Petamo GHY 133 N has a stable consistency up to about 9 MGy, whereas the others become fluid below 5 MGy.

Petamo GHY 133 N and RG-42R-1 have a comparable stability with dose, despite their different chemical composition. The former is realized with a mineral oil and a polyurea thickener, while the latter is realized with polyphenylether oil and a polycarbonate thickener.

Table 5
Thresholds for the tested greases associated to the functional endpoint definition, corresponding to a 10% variation from the original consistency. Thresholds are determined using a linear interpolation. When variations are lower than 10% for all the tested dose values, consistency variation at the maximum dose is reported.

Product	Dose MGy	Cons. var %	Fluence (n) par cm^{-2}	Fluence (γ) par cm^{-2}
Turmopolgrease 2	0.1	+10%	7.6 10^{15}	7.3 10^{15}
Krytox 240 AC	0.2	+10%	4.1 10^{16}	4.0 10^{16}
Grizzlygrease No. 1	0.3	+10%	2.2 10^{16}	2.1 10^{16}
AFB-LF	0.4	+10%	2.7 10^{16}	2.6 10^{16}
FAG Arcanol LOAD 220	1.1	+10%	7.5 10^{16}	7.2 10^{16}
Klüberlub BE 41-542	1.8	+10 %	1.2 10^{17}	1.2 10^{17}
Apiezon M	2.3	+10%	1.5 10^{17}	1.5 10^{17}
RG-42R-1	7.6	+5%	6.2 10^{17}	5.9 10^{17}
Petamo GHY 133 N	8.9	−3%	6.2 10^{17}	5.9 10^{17}

Therefore, it is concluded that the chemical composition of the oil alone is not representative of the grease radiation sensitivity. Specific additives are expected to influence radiation stability as well. For those reasons, general conclusions based on the chemical composition can be hardly drawn and specific products must be individually tested.

4.3. Thresholds of radiation damage

The concept of radiation damage threshold in greases is scarcely defined. However, it is of utmost importance in the engineering viewpoint and for end-users like the ESS and the SPES facilities. In the present work, two endpoints are defined to evaluate the radiation-resistance of the tested products.

A 10% deviation from the non-irradiated consistency is considered in the present study as a functional endpoint. Consistency can be expressed as well in NLGI grades, ranging from 000 (soft) to 6 (hard). The NLGI grade is a relevant parameter for the grease specific application. A deviation of one grade from the original consistency grade approximately corresponds to a 10% consistency variation. Table 5 contains the thresholds associated to this functional endpoint. Thresholds are expressed in terms of total absorbed dose and in terms of exposure to the total neutron and gamma fluence in the mixed irradiation field of the facility.

A severe structural endpoint is associated to the radiation-induced fluidization of the grease. It corresponds to consistency values being out of the maximum penetrometer range. This endpoint is evidenced by dashed lines in Fig. 8 and marks an end of life condition for the irradiated grease.

The defined endpoints provide useful dose and particle fluence thresholds to compare the products radiation-resistance. Petamo GHY

133 N and RG-42R-1 are identified as the most radiation resistant products comparatively. In fact, their consistency deviation from the original value is lower than 10% for all the investigated dose values. For these products, the consistency variation at the maximum absorbed dose is reported in Table 5. Fluorinated products, despite the lower amount of neutron dose absorbed in comparison with hydrogenated ones, are not recommended for application in radioactive environments. In fact, the acid gases evolved during irradiation could damage the surrounding metallic components.

4.4. Comparison with producers' declarations

Consistency results obtained in the present work can be compared with the radiation resistance declarations of the producers. The results achieved on Grizzlygrease No.1 are particularly intriguing. Its consistency abruptly increases at 0.4 MGy of mixed neutron and gamma dose. This value is much lower than the 1.2 MGy gamma dose threshold declared by the producer. The equivalence of the effects of gamma dose and neutron dose in polymers has been longly believed. However, the differences reported for Grizzlygrease No.1 could be attributed to the different irradiation condition adopted in the tests, in particular the use of pure gamma or mixed radiation fields.

In addition, it is interesting to note that the two most stable greases have different producer declarations concerning the radiation resistance. RG-42R-1 is declared by the producer as radiation resistant up to 15 MGy of gamma dose, while Petamo GHY 133 N has no radiation resistance declaration.

It can be concluded that the products lacking a radiation resistance declaration do not necessarily feature scarce radiation resistance. In fact, only a limited number of companies in the world tested their products with radiation. As here reported, some greases featuring excellent performance but not tested with radiation show excellent radiation stability. On the contrary, products having a declared dose threshold do not necessarily have a comparable radiation resistance when irradiated at same doses in different radiation fields. This evidence supports the need for further testing on specific products, especially when it is needed to know their radiation resistance in specific irradiation conditions. The producer's declaration based on gamma irradiation cannot be considered as satisfactory for the use of a product in a different radiation field.

4.5. Predictive ability of the results

In the present study, greases are irradiated in a neutron and gamma mixed reactor field. The chosen irradiation conditions are significant for applications in facilities that will produce similar radiation fields, as the SPES and the ESS systems. However, the radiation fields in operation will differ from the one used in the present study by several parameters. Firstly, the dose rate expected in operation will be in general orders of magnitude lower than the one used for testing in the reactor. Tests in the reactor are necessarily accelerated. Greases have been irradiated in reactor up to several MGy of total dose, corresponding to neutron and gamma fluences of the order of magnitude of 10^{17} particle cm^{-2}. Similar exposures will be cumulated in much longer operation times, ranging from weeks to decades in the SPES and in the ESS facilities. Testing conditions can not easily replicate so long exposure times, especially in case of extensive experimental campaigns which count several products irradiated at various dose levels. To complete irradiation in a reasonable time, quicker radiation damage tests, referred to as accelerated, are necessary.

Moreover, greases are tested in air atmosphere at atmospheric pressure, while in some applications they will work in vacuum or in absence of oxygen. The temperature in the irradiation facility is higher that the temperature expected for most applications. The expected neutron spectrum in operation will differ from the reactor one. Neutrons with higher energy will be produced by spallation neutron targets and by facilities for the production of radioactive ion beams.

Damage mechanisms in polymers are expected to depend on several parameters as the radiation fields, the dose rate, the oxygen diffusion in the material and the temperature, in a synergistic way. For this reason, the present results can not be used to exactly predict the expected grease behaviour in specific operating conditions. Nevertheless, the consistency behaviour of several different commercial greases irradiated in the same conditions in a neutron and gamma mixed field over a total absorbed dose of about 10 MGy are here reported. The set of collected data is unique in the present literature.

5. Summary and conclusions

The present work approaches the problem of radiation effects on lubricating greases from an experimental viewpoint. A methodology for grease irradiation in a neutron and gamma mixed field and post-irradiation examination is developed. Nine commercially available products are selected from some of the most important producers in the world. Grease samples are irradiated in an in-core facility of a research nuclear reactor at absorbed doses ranging between 0.1 MGy and 9 MGy. Dosimetry in the neutron and gamma mixed field is calculated. Consistency is tested after irradiation as the most significant functional parameter characterizing a grease. Extreme radiation-induced consistency modifications are observed for seven out of the nine tested products, leading to the grease fluidization. Two out of the nine tested products exhibit stable consistency as a function of the dose.

It can be concluded that the cleavage of the grease structure is the dominant process in the chosen irradiation conditions. This is evidenced by the grease fluidization and by the evolution of gas, which depends on the thickener disruption and on the production of fragments respectively.

Some of the greases having base oils with a similar chemical composition show very different radiation effects. For this reason, it is not possible to directly correlate the chemical structure of greases with their radiation resistance. The present study contributes to provide experimental evidences of cleavage as the dominant process induced by radiation on the grease structure up to the tested values of absorbed dose. However, the collected data evidence the need for a further understanding of the interaction between radiations and the complex multi-phase chemical structure of greases.

End of life usability conditions are presented to propose radiation thresholds for the greases. Two greases are comparatively evaluated as the most radiation resistant ones. Some of the results allow the commonly used definitions of radiation-resistance to be questioned. In fact, some of the commercial products which are declared by the producer as radiation resistant, show extreme degradation in the present study. These producer declarations refer to gamma radiation only. The observed differences are probably due to the different irradiation conditions, which influence the radiation damage. For this reason, gamma radiation data should not be used to draw general conclusions on the radiation resistance of greases and to define universally accepted dose thresholds. In addition, the presented results challenge the generally believed assumption claiming that equal doses of different radiations are equally effective in damaging polymers. The use of mixed radiation fields is necessary to more accurately test the resistance of polymeric materials that must work in the presence of mixed radiations.

The results collected in this paper represent a set of data achieved in irradiation conditions that differ from the ones commonly employed to test polymers. The developed methodology here described allows the radiation resistance of the tested greases to be comparatively evaluated. Apart from the scientific significance, the data collected in mixed neutron and gamma radiation are particularly relevant for new generation facilities in which the used greases are exposed to a mixed neutron and gamma environment. Discussions with some of the grease providers are ongoing as well for a deeper investigation and understanding of the basic mechanisms that determine the observed phenomena. Chemical analyses on selected irradiated greases will be performed for a deeper understanding of the experimental results presented in this paper.

Declarations

Author contribution statement

M. Ferrari: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

A. Zenoni, Y. Lee: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

M. Hartl: Analyzed and interpreted the data.

A. Andrighetto: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

A. Monetti: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

A. Salvini: Contributed reagents, materials, analysis tools or data.

F. Zelaschi: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

Data availability

The raw data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study. The processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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