Effects of 400 keV electrons flux on two space grade silicone rubbers

Hélène Jochem, Virginie Rejsek-Riba, Eddy Maerten, Stéphanie Remaury, Stéphane Solé, Guillaume Sierra, Antoine Baceiredo, Olivier Guillaumon

► To cite this version:

Hélène Jochem, Virginie Rejsek-Riba, Eddy Maerten, Stéphanie Remaury, Stéphane Solé, et al.. Effects of 400 keV electrons flux on two space grade silicone rubbers. *Materials Chemistry and Physics*, 2013, 141 (1), pp.189-194. 10.1016/j.matchemphys.2013.05.002 . hal-03412860

HAL Id: hal-03412860

<https://hal.science/hal-03412860>

Submitted on 3 Nov 2021

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

**Elsevier Editorial System(tm) for Materials Chemistry
and Physics Manuscript Draft Manuscript Number:
MATCHEMPHYS-D-12-00453R2 Title: Effects of 400
keV electrons flux on two space grade silicone rubbers
Article Type: Full Length Article**

Hélène Jochem, Virginie Rejsek-Riba, Eddy Maerten, Stéphanie Remaury,
Stéphane Solé, Guillaume Sierra, Antoine Baceiredo, Olivier Guillaumon

► **To cite this version:**

Hélène Jochem, Virginie Rejsek-Riba, Eddy Maerten, Stéphanie Remaury, Stéphane Solé, et al.. Elsevier Editorial System(tm) for Materials Chemistry and Physics Manuscript Draft Manuscript Number: MATCHEMPHYS-D-12-00453R2 Title: Effects of 400 keV electrons flux on two space grade silicone rubbers Article Type: Full Length Article. Materials Chemistry and Physics, Elsevier, 2013. hal-03412860

HAL Id: hal-03412860

<https://hal.archives-ouvertes.fr/hal-03412860>

Submitted on 3 Nov 2021

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Manuscript Number: MATCHEMPHYS-D-12-00453R2

Title: Effects of 400 keV electrons flux on two space grade silicone rubbers

Article Type: Full Length Article

Keywords: Silicone rubber; Electron irradiation; Chemical structure degradations; Mechanical and Thermo-optical properties

Corresponding Author: Dr. Eddy Maerten, Ph.D

Corresponding Author's Institution: Universite Paul Sabatier

First Author: Hélène Jochem

Order of Authors: Hélène Jochem; Virginie Rejsek-Riba, Ph.D; Eddy Maerten, Ph.D; Stéphanie Remaury, Ph.D.; Stéphane Solé, Ph.D; Guillaume Sierra, Ph.D; Antoine Baceiredo, Ph.D; Olivier Guillaumon

Abstract: Two different space grade silicone rubbers were irradiated by an electron flux of 400 keV. The irradiation impact strongly depends on the chemical structure of rubbers (one reinforced with MQ resins, and the other one functionalized with phenyl groups at the silicon atoms and reinforced with silica). The irradiated rubbers were studied by means of solvent swelling, solid-state ^{29}Si NMR, and ATR - FTIR spectroscopy. Physical properties were evaluated by thermal (differential scanning calorimetry), mechanical (dynamic mechanical analysis), and thermo-optical (ultraviolet-visible-near infrared spectroscopy) analyses. The formation of silicium T units and Si-CH₂-Si networks were evidenced by ^{29}Si NMR, and the increase of the glass transition temperature and of modulus reflect the substantial increase in the macromolecular chain rigidity of the irradiated material. Dramatic damages of mechanical properties were observed, depending on the reinforced materials used. Slight changes of thermo-optical properties were highlighted independently to the initial chemical structure.

Fig. 1. Hydrosilylation reaction between vinyl silicone MQ resins and silicone functionalized chains

Fig. 2. Silica dispersed in the network (left) / MQ resins inside the network (right)

Fig. 3. Standard nomenclature for siloxane polymers (with R = -CH₃ /-H /-C₂H₅...)

Fig. 4. ²⁹Si NMR spectra of pristine 2D (left) and 3D rubbers (right) (* correspond to rotation band)

Fig. 5. ²⁹Si NMR spectra of irradiated 2D (left) and 3D rubbers (right)(* correspond to rotation band)

Fig. 6. ATR-FTIR spectra of pristine and irradiated 3D rubber

Fig. 7. ATR-FTIR spectra of pristine and irradiated 2D rubber

Fig. 8. Cross-linking by T siloxane units formation

Fig. 9. Cross-linking by Si-CH₂-Si siloxane units formation

Fig. 10. -SiH groups formation

Fig. 11. Storage modulus (MPa) of pristine and irradiated rubbers

Fig. 12. Diffuse transmission in the range 200 to 600 nm of pristine and irradiated rubbers

Table 1. Assignment of pristine rubbers NMR peaks

Table 2. Average Molecular weight between cross-links (Mc) of pristine and irradiated rubbers

Table 3. Glass transition temperatures of pristine and irradiated rubbers

*Highlights

Electron flux radiations generate chains cross-linking.

Cross-linking occurs by T and Si-CH₂-Si units formation.

The cross-linking rate is slightly faster for 2D rubber compared to 3D rubber.

Modulus increases much more significantly for 3D rubber.

Effects of 400 keV electrons flux on two space grade silicone rubbers

H. Jochem^{1,2,3}, V. Rejsek-Riba¹, E. Maerten^{2,*}, S. Remaury³, S. Solé⁴, G. Sierra⁴, A. Baceiredo², O. Guillaumon⁴

¹ONERA - The French Aerospace Lab F-31055 Toulouse, France

²Université de Toulouse, UPS, and CNRS, LHFA, UMR 5069, 118 route de Narbonne, F-31062 Toulouse cedex 9, France

³CNES -French Aerospace Agency – 18 avenue Edouard Belin F-31401 Toulouse cedex 9, France

⁴MAP Coatings - ZI - 2 Rue Clément Ader 09100 Pamiers, France

Corresponding Author:

Eddy Maerten

Universite Paul Sabatier

LHFA, Batiment 2R1, 2eme etage

118, route de Narbonne

31062 Toulouse cedex 9

France

Mail : maerten@chimie.ups-tlse.fr

Phone : (33) 5 61 55 64 03

Fax : (33) 5 61 55 82 04

Abstract

Two different space grade silicone rubbers were irradiated by an electron flux of 400 keV. The irradiation impact strongly depends on the chemical structure of rubbers (one reinforced with MQ resins, and the other one functionalized with phenyl groups at the silicon atoms and reinforced with silica). The irradiated rubbers were studied by means of solvent swelling, solid-state ²⁹Si NMR, and ATR – FTIR spectroscopy. Physical properties were evaluated by thermal (differential scanning calorimetry), mechanical (dynamic mechanical analysis), and thermo-optical (ultraviolet-visible-near infrared spectroscopy) analyses. The formation of silicium T units and Si-CH₂-Si networks were evidenced by ²⁹Si NMR, and the increase of the glass transition temperature and of modulus reflect the substantial increase in the macromolecular chain rigidity of the irradiated material. Dramatic damages of mechanical properties were observed, depending on the reinforced materials used. Slight changes of thermo-optical properties were highlighted independently to the initial chemical structure.

Keywords: Silicone rubber; Electron irradiation; Chemical structure degradations; Mechanical and Thermo-optical properties

1. Introduction

Due to their excellent properties such as good resistance to oxidation and ultraviolet radiations, wide operating temperature range, excellent insulating properties [1-3] silicone rubbers are used on spacecraft such as thermal-control coatings or adhesives for solar cells. In geostationary orbit (GEO), the spacecraft is exposed to space environment mainly characterized by high vacuum, Ultra-Violet (UV) radiations and charged particles (electrons and protons). This aggressive environment causes degradation of materials [4,5]. Therefore, it is necessary to study and understand space environment effects on the chemical silicone structure and to evaluate mechanical and thermo-optical properties in order to forecast spacecraft life-time. Many studies have been conducted on the effects of UV and protons radiations. UV radiations are known to be responsible for thermo-optical properties degradations [6-8]. Protons radiations cause mechanical properties degradations and surface cracks [9-12]. However, only a few studies were focused on the damages caused by electrons radiations [13,14] and were interested in the correlation between chemical structure degradations and technological properties evolution.

The aim of this work is to study the electron radiations effects on two space grade silicone rubbers with different chemical structures, prepared by polyaddition reactions. The first one contains silicone MQ resins as reinforced material, whereas the second one, functionalized with phenyl groups at the silicon atoms, is silica-filled. The irradiated materials were studied by means of solvent swelling, solid-state ^{29}Si NMR, and Attenuated Total Reflection (ATR) Fourier Transform Infrared (FTIR) spectroscopy. The evolution of physical properties, mechanical and thermo-optical, was evaluated by differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), and ultraviolet-visible-near infrared (UV-Vis-NIR) spectroscopy.

2. Experimental

2.1. Materials

Silicone rubber containing MQ resins as reinforced materials was prepared by hydrosilylation catalyzed by Karstedt complexe $\{\text{Pt}_2[\text{O}(\text{Si}(\text{CH}_3)_2(\text{CH}=\text{CH}_2))_2]_3\}$ from Dow Corning (DC) 93-500 Space Grade Encapsulant two part silicone elastomer kit (Fig. 1). The curing agent was used at a ratio 1/10 (w/w). Silicone rubber containing silica as reinforced material was prepared from Wacker Elastosil S 690 kit. The curing agent was used at a ratio 1/1 (w/w). After mixing the base and the curing agent, two sample types were prepared. Thin layers of rubber were successively deposited on quartz substrates until reaching 150 μm thickness by manual way. For the second type of sample, the solution's kit was poured in a mould of 500 μm depth to obtain 500 μm thickness films, and then air degassed. Finally, polymers were cured during 16 h at 65 $^\circ\text{C}$. Thin coatings on quartz substrates were prepared to measure thermo-optical properties. 500 μm thickness films were prepared to perform chemical analyses and evaluate mechanical properties.

Throughout this paper, the silicone rubber containing silicone MQ resins will be named "3D rubber", and the silica-filled silicone rubber will be named "2D rubber" (Fig. 2).

2.2. Irradiation

Samples were placed on a sample holder in a vacuum vessel (ONERA facility) connected to a 2.7 MeV Van de Graaff accelerator supplying electrons beam. After 16 h pumping, a vacuum of 10^{-6} mbar was reached. The electron energy was 400 keV and the irradiation current was fixed at 20 nA/cm². The fluence was 3.10^{15} electrons/cm² corresponding to 3 years in GEO for the satellite North / South faces. After irradiation, samples were analysed under air conditions.

2.3. NMR spectroscopy

Solid-state ²⁹Si NMR spectra were recorded on a Bruker Avance 400 spectrometer equipped with a 4 mm probe operating at 79.39 MHz. Samples were spun at 12 kHz at the magic angle using ZrO₂ rotors. ²⁹Si MAS (magic angle spinning) spectra were obtained under high-power proton decoupling conditions; with a small flip angle (30°) and a recycle delay of 60 s.

2.4. ATR-FTIR spectroscopy

The ATR-FTIR spectra were collected using a Nicolet 800 FT-IR spectrometer equipped with a deuterated triglyceride sulfate detector (DTGS) and coupled with a diamond ATR accessory.

2.5. Swelling tests

Toluene was used as solvent in these tests. Silicone rubber samples were submerged in toluene, and the swollen weight was recorded periodically until an equilibrium weight was obtained (about 3 days). Just before weighting, the solvent on the surface was absorbed off quickly with filter paper. The samples were dried at room temperature during 7 days in air and reweighted for the final dry weight. The molecular weight between crosslinks M_c was then calculated using the Flory–Rehner relationship [15,16]:

$$M_c = \frac{V_{s, mol} \cdot \rho \cdot \left(\frac{\Phi}{2} - \Phi^{1/3} \right)}{\ln(1 - \Phi) + \Phi + \chi \cdot \Phi^2}$$

where ρ is the density of the rubber network (g/ cm³) , Φ the volume fraction of the polymer in the swollen mass, $V_{s, mol}$ the molar volume of solvent (106.3 mL / mol for toluene) and χ the Flory- solvent-polymer interaction parameter (0.791 for the system PDMS-toluene) [17].

2.6. DMA and DSC

Dynamic mechanical analysis (DMA) was used to determine the storage modulus E' and Differential Scanning Calorimetry (DSC) to determine the glass transition temperature. DMA assays were carried out on a Mettler Toledo DMA/ SDTA 86 instrument operating on traction mode, at a frequency of 1 Hz at room temperature, using samples of 0.5 x 1.9 x 2 mm³.

The DSC measurements were performed on a Mettler Toledo differential scanning calorimeter (model DSC 1 STARe system) under dry nitrogen. An empty aluminium pan was used as reference. In the first run, the sample is heated from room temperature to 150 °C, then cooled to -140 °C. Data were recorded during the second run for temperatures ranging from -140 °C to 150 °C at a heating rate of 10 °C.min⁻¹. The cooling rate between the first and second run was also equal to 10 °C.min⁻¹. The glass transition temperature (T_g) was calculated at the inflection point of the transition.

2.7. UV-Vis-NIR spectroscopy

The UV-Vis-NIR measurement system of ONERA is composed of a Perkin Elmer Lambda 900 spectrophotometer connected to an integrating sphere in air. The calibration was performed using a Spectralon SRS-99-010 standard. The diffuse transmission spectra in the range 200-2500 nm were acquired at intervals of 1 nm, with the lamp change at 319.2 nm.

3. Results and Discussion

3.1. ²⁹Si NMR

The pristine rubbers were characterized by ²⁹Si NMR. In order to make an easier spectra description, the standard shorthand nomenclature will be used to identify molecular structures (Fig. 3) as following [18]:

The ²⁹Si NMR spectra of pristine rubbers are shown in Fig. 4 and peak assignments are summarized in Table 1 [19,20]. Spectra have three peaks in common at - 22 ppm assigned to D siloxane units within the chain, - 109 / - 110 ppm attributed to Q motifs and 7.5 ppm attributed to M siloxane units at the polymer chain ends. However, some differences can be observed. The peak at - 109 / - 110 ppm is broad for 2D rubber and well-defined for 3D rubber corresponding to silica and Q units in MQ resins respectively. 3D rubber spectrum presents another peak at 12 ppm which can be assigned to M units in MQ resins and a peak at - 37 ppm attributed to Si-H groups inside the chains. 2D rubber spectrum shows an intense peak at - 48 ppm attributed to Si-phenyl functions along the chains [21].

After irradiation, a new peak at - 67 ppm appears for each rubber, which can be assigned to T siloxane units (Fig. 5). In the case of 3D rubber a small signal at - 28 ppm can be assigned to Si-CH₂-Si cross-link unit as described for gamma irradiations [20,22]. The signal was not observed in the case of 2D rubber, which contains less ≡Si-CH₃ groups than 3D rubber.

3.2. ATR-FTIR

ATR - FTIR spectra, in the range 1300-600 cm⁻¹, of irradiated rubbers compared with the pristine ones are presented in Fig. 6 and 7. For both irradiated-rubbers, only changes in the vibrational band intensity were observed. In the case of 3D rubber, an increase of the absorption band at 906 cm⁻¹ is observed which could be attributed to an increase of the number of Si-H groups. The peaks at 786 and 1257 cm⁻¹ assigned to Si-CH₃ groups decrease in intensity. However an increase of the peaks at 842 and 756 cm⁻¹, attributed to methyl groups at the chain ends [-Si(CH₃)₃], is observed for both irradiated rubbers. Finally, slight

changes of the broad band between 1000-1130 cm^{-1} are ascribed to modification of Si-O-Si bonds.

From NMR and ATR-FTIR analyses, it can be postulated that electrons radiations cause the formation of radicals: $\equiv\text{Si}^\bullet$, $-\text{CH}_2^\bullet$, $\text{CH}_3^\bullet$, and $\text{H}^\bullet$. Then crosslinks are formed by recombination of polymer radicals through T and Si-CH₂-Si units (Fig. 8 and 9), and side-chain functionalization by M motifs and Si-H groups were also observed (Fig. 10). Phenyl groups on silicone chains seem privileged the formation of T and M motifs.

3.3. Solvent swelling

Cross-linking evidenced by NMR analysis should be corroborated by an increased network density. To confirm these results, swelling tests were carried out on pristine and irradiated rubbers (Table 2). Average molecular weights between cross-links (M_c) for pristine and irradiated rubbers are different. Before irradiation, M_c of 2D rubber is 2.5 times higher than 3D rubber one. After irradiation, the M_c values have decreased dramatically highlighting an increase of cross-link density, in agreement with NMR results. The cross-linking rate is slightly faster in the case of 2D rubber despite the presence of phenyl groups along silicone chains known to reduce remarkably the cross-linking efficiency [23]. Cross-linking is probably favoured in this rubber because of the initial low density of the network, resulting in high chain mobility. On the contrary, in the case of 3D rubber, the cross-linking would be disfavoured by the high initial density network.

3.4. DSC

Glass transition temperature values (T_g) are presented in Table 3. The phenyl groups along the chain in pristine and 2D rubber induce a higher T_g than in 3D rubber [24].

After irradiation, T_g have increased by about 5 °C for both rubbers. It traduces a decrease of chains mobility due to an increase of cross-link density, in agreement with the reduction of chain length between cross-links.

3.5. DMA

Fig. 11 illustrates storage modulus E' (MPa) of pristine and irradiated rubbers. Irradiation leads to an increase in modulus for both rubbers, and to a degradation of mechanical properties. The modulus variation is much more significant for 3D rubber compared to 2D one. According to solvent swelling and DSC results, this difference can not be only related to the cross-linking rate. Although, this could be explained at the network architecture level. Indeed, for 3D rubbers, the pristine network is not fully cross-linked (pending vinyl groups on MQ resins), and, during electron irradiation, cross-linking through these reactive sites can be expected to give an increase of the rigidity. Whereas in 2D rubber, cross-linking seems to occur predominately between chains, leading to a more flexible network.

3.6. UV-Vis-NIR spectroscopy

UV-Vis-NIR spectra of pristine and irradiated rubbers are presented in Fig. 12. Before irradiation, the UV-cut off wavelengths, at 75 % of transmission, are 230 nm and 280 nm for 3D and 2D rubbers respectively. Silica and phenyl groups in 2D rubber absorb at wavelengths below 280 nm and explain a higher UV-cut off. After electron flux irradiations, the UV-cut off evolution is similar for both rubbers with a slope decrease and **shift** to higher wavelengths. The UV-cut off wavelengths, at 75 % of transmission, are now 300 nm and 340 nm for 3D and 2D rubbers respectively. The degradation rate seems to be similar for both rubbers independently **of** the initial chemical silicone structure. The UV cut off wavelengths are situated below 400 nm, so no yellowing is observed and the thermo-optical properties degradation caused by electrons radiations can be considered non- significant. It can be supposed that the slight degradation of transmission in both rubbers is due to the common chemical degradations.

4. Conclusion

Electron flux radiations cause substantial degradation in mechanical properties, whereas thermo-optical properties are only slightly damaged. In particular, electron flux radiation-induced cross-linking of silicone rubbers significantly increases network density, glass transition temperature, and modulus. This phenomenon mainly occurs *via* silicium-T and Si-CH₂-Si units formation. Side-chain functionalization by Si-H groups and M siloxane units were also observed. The cross-linking rate is slightly faster for 2D rubber compared to 3D rubber, whereas modulus increase is much more significant for 3D rubber. MQ resins in 3D rubber are probably responsible of the very high increase of modulus. In conclusion, 2D rubber are probably more suitable than 3D rubber for applications requiring mechanical properties preservation.

References

- [1] W. Noll, Chemistry and Technology of silicones, Academic Press, New York, 1968.
- [2] M. Biron, Silicones ou siloxanes- structure et propriétés, Techniques de l'Ingénieur (2007), N 2280.
- [3] F. De Buyl, J. Adhesions Adhesives, 21 (2001) 411-422.
- [4] J. Marco, S. Remaury, High Perform. Polymer, 16 (2004) 177 – 196.
- [5] J. Marco, C. Pons C, Simulation of 1 year GEO orbit. Intermediate report (2006), unpublished results.
- [6] E. Grossman, I. Gouzman, Nucl. Instr. Meth. Phys. Res. B 208 (2003) 48-57.
- [7] J. A. Dever, B. A. Banks, L. Yan, Space Technology Proceeding. 6 (2006) 123-140.
- [8] J. Marco, C. Pons, UV and Particles Irradiations on Thermal Control Coatings for Simulated Space Environment, Final Report (2002).
- [9] L. X. Zhang, Sh. Y. He, Zh. Xu, Q. Wei, Mater. Chem. Phys. 83 (2004) 255-259.
- [10] L. Zhang, Zh. Xu, Q. Wei, Sh. He, Radiat. Phys. Chem. 75 (2006) 350–355.
- [11] D. Mingwei, Sh. He, R. Li, D. Yang, Nucl. Instr. Meth. Phys. Research B 248 (2006) 31-36
- [12] C. Whang, J. Spacecrafts and Rockets, 43 (2006) 520-522.
- [13] I. Stevenson, L. David, C. Gauthier, L. Arambourg, J. Davenas, G. Vigier, Polym. 42 (2001) 9287-9292.
- [14] M. Frounchi, S. Dadbin, F. Panahinia, Nucl. Instr. Meth. Phys. Research B 243 (2006) 354-358.
- [15] P. J. Flory , J. Rehner, J. Chem. Phys. 11 (1943) 512.
- [16] P. J. Flory , J. Rehner, J. Chem. Phys. 11 (1943) 521.
- [17] M. Braden, D. Latham, M. P. Pathel, Eur. Polym. J. 41 (2005) 3069-3071.
- [18] C. B. Hurd, J. Am. Soc. 68 (1946) 364-370.
- [19] E. A. Williams, J. D. Cargioli, Annu. Rep. NMR Spectrosc. 9 (1979) 221.
- [20] D. J. T. Hill, C. M. L. Preston, A. K. Whittaker, Polym. 43 (2002) 1051-1059.
- [21] R. A. Newmark, B. C. Copley Macromol. 17 (1984) 1973-1980.
- [22] A. Charlesby, Proc. Roy. Soc. A, 215 (1955) 187.
- [23] C. G. Deldies, Radiat. Phys. Chem. 16 (1980) 345-352.
- [24] G. N. Babu, S. S. Christopher, R. A. Newmark, Macromol. 20 (1987) 2654-2659.

M D T Q

Table 1. Assignment of pristine rubbers NMR peaks

Peak	2D rubber	3D rubber
7.5 ppm	M siloxane unit at the end of polymer chains	
– 22 ppm	D siloxane unit	
12 ppm	-	M siloxane unit in MQ resin
– 109/ – 112 ppm	Q siloxane unit	
– 37 ppm	-	D ^H siloxane unit
– 48 ppm	D ^{phenyl} siloxane unit	-

Fig. 4. ^{29}Si NMR spectra of pristine 2D (left) and 3D rubbers (right) (* correspond to rotation band)

Fig. 5. ^{29}Si NMR spectra of irradiated 2D (left) and 3D rubbers (right) (* correspond to rotation band)

Fig. 6. ATR-FTIR spectra of pristine and irradiated 3D rubber

Fig. 7. ATR-FTIR spectra of pristine and irradiated 2D rubber

Fig. 8. Cross-linking by T siloxane units formation

Fig. 9. Cross-linking by Si-CH₂-Si siloxane units formation

Fig. 10. -SiH groups formation

Table 2. Average Molecular weight between cross-links (Mc) of pristine and irradiated rubbers

	Mc (g/mol) Pristine sample	Mc (g/mol) Irradiated sample	Ratio (Mc pristine/ Mc irradiated)
3D rubber	1330	230	5.7
2D rubber	3300	400	8.3

Table 3. Glass transition temperatures of pristine and irradiated rubbers

	Tg (°C) Pristine sample	Tg (°C) Irradiated sample
3D rubber	-121.0	-116.3
2D rubber	-115.0	-110.3

Fig. 11. Storage modulus (MPa) of pristine and irradiated rubbers

Fig. 12. Diffuse transmission in the range 200 to 600 nm of pristine and irradiated rubbers

Figure 1 revised
[Click here to download high resolution image](#)

M

D

T

Q

Peak	2D rubber	3D rubber
7.5 ppm	M siloxane unit at the end of polymer chains	
— 22 ppm	D siloxane unit	
12 ppm	-	M siloxane unit in MQ resin
— 109/— 112 ppm	Q siloxane unit	
— 37 ppm	-	D ^H siloxane unit
— 48 ppm	D ^{phenyl} siloxane unit	-

	Mc (g/mol) Pristine sample	Mc (g/mol) Irradiated sample	Ratio (Mc pristine/ Mc irradiated)
3D rubber	1330	230	5.7
2D rubber	3300	400	8.3

	Tg (°C) Pristine sample	Tg (°C) Irradiated sample
3D rubber	-121.0	-116.3
2D rubber	-115.0	-110.3

Figure 1 BnW revised

[Click here to download high resolution image](#)