SCANNING ELECTRON MICROSCOPY ANALYSIS OF ELASTOMERIC O-RINGS AND SPRING SEALS FRACTURED DURING RAPID GAS DECOMPRESSION.

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Abstract

Rapid Gas Decompression (RGD), commonly known as explosive decompression (ED) is an operational condition during which applied system pressure is quickly released, resulting in the expansion of absorbed gasses damaging elastomeric seals. This research investigated fracture on two types of elastomeric seals exposed to RGD conditions, using fractography methods. Fractured Fluorocarbon elastomer O-rings and Hydrogenated Nitrile Butadiene Spring seals surfaces were examined after being exposed to Rapid Gas Decompression conditions. Scanning Electron Microscopy analyses revealed that small cracks initiated from micro-voids in the centre region of the O-ring and propagated towards the inner and outer circumference of the O-ring. When the initial crack reached a critical size, the crack propagation rapidly accelerated resulting in a catastrophic failure. All fracture surfaces displayed a more pronounced relatively stable crack growth towards the inner circumference of the O-rings. The fracture surfaces contained pits on the surface and these pits were a result of reinforcing agglomerates coming out of the matrix.

1. Introduction

Rapid Gas Decompression (RGD), commonly known as explosive decompression (ED), is an operational condition during which applied system pressure is quickly released, resulting in the expansion of absorbed gas damaging elastomeric seals, Briscoe [1]. In service, O-rings and spring seals are exposed to gasses at high pressure. Gasses in contact with the seal surface diffuse into the elastomer until the material is fully saturated. At high pressure, the absorbed gas is in a compressed state and when the external pressure is suddenly released the compressed gas expands at a faster rate than it can naturally diffuse through the elastomer. If the material contains voids or rigid inclusions, the compressed gas nucleates at these sites and during rapid gas decompression the voids inflate resulting in tensile stresses or strains in the void walls. If the stresses are higher than the strength of the elastomer, cracks initiate and propagate.

To avoid failure of sealing components in operation, there are several industry and customer specific test methods used for testing elastomeric O-rings exposed to RGD conditions. These tests differ from one another in a number of aspects and hence, the suitability of a particular material proven under a particular test regime may not necessarily guarantee it's suitability if tested under another test. Some of the common test procedures are:

i. Norsok Standard M-710 Rev.2, October 2001 " Qualification of non-metallic sealing materials and manufacturers"
ii. ISO 23936-2:2011 " Petroleum and natural gas industries – Non-metallic materials in contact with media related to oil and gas production" [2]
iii. SHELL test procedure, as described by Cox, [3].
iv. TOTALFINA SP-TCS-142 APPENDIX H "Elastomer "O"-ring Seals Explosive Decompression Type Testing Procedure [4].

The test procedure used in this investigation is the Norsok M-710 Rev.2 [5] test regime. The Norsok standards were developed by a Norwegian petroleum industry and they define the requirements for critical elastomer sealing, seat and back up materials for permanent use subsea.

2. Method

Three fractured Fluorocarbon FKM type 3 O-ring surfaces were examined after being exposed to Rapid Gas Decompression according to the Norsok M-710 rev.2, 2001 Annex B standards. Table 1 shows the conditions in which the three O-rings were tested.

Compound	Test Pressure (bar)	Test Temperature (^{O}C)	Test Conditions
FKM type 3	600	100	90% CO_2 in methane
FKM type 3	600	100	90% CO_2 in methane
FKM type 3	400	100	70% CO_2 in methane

Table 1: showing the O-rings testing conditions during rapid gas conditions

The pressure was released at a rate of 20 bars per minute during each cycle and each O-ring was subjected to 10 decompression cycles according to the Norsok M710 Rev2 standard. The spring seals were tested according to ISO 23936 – 2 standards at a temperature of 120^{o}C. Fractured surfaces of both O-ring and spring seals are non-conductive and therefore they are required to be either carbon coated or gold coated. In this investigation the fractured surfaces were carbon coated in case elemental analysis needed to be conducted on the fractured surfaces. The fractured surfaces were then analysed using a scanning electron microscope (JOEL 6400).

3. Results

After conducting R.G.D. tests, the fractured surfaces were analysed using SEM analysis to identify the initiation points and the mode of crack propagation. Fig. 1 displays two distinct regions of fracture which are the initiation regions A and B and the rapid crack propagation region C. Cracks initiate in region A and B then propagate in slow increments during each loading cycle. When the crack reaches a critical size, the crack becomes unstable and grows at a faster rate leading to a catastrophic macroscopic featureless fracture displayed in the region C. The initiation region is characterised by many small tearing lines obstructing one another and the fracture surface is microscopically rough. The crack propagation direction can be identified from the direction in which riverlines converge and the crack propagation direction in Fig. 1 is shown by the arrows.

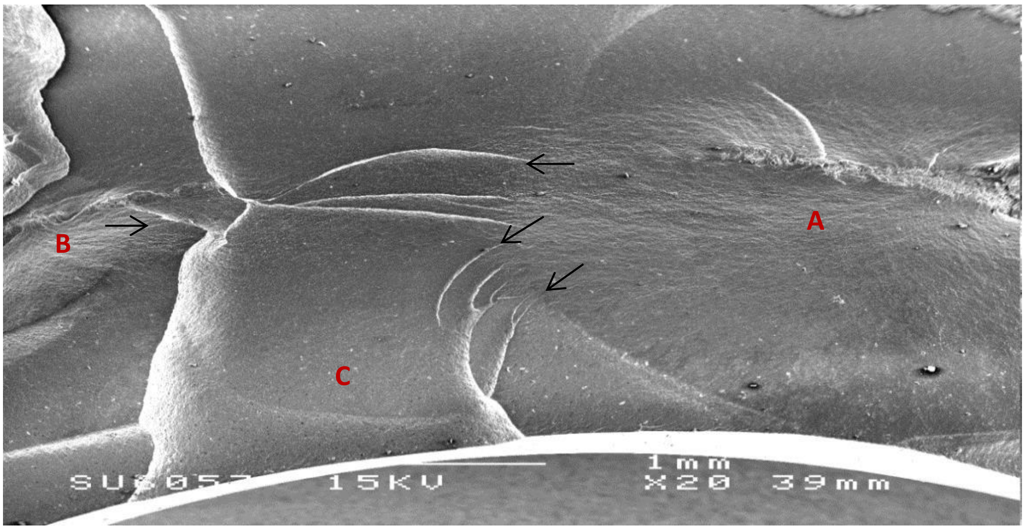

Fig.1 SEM showing fractured surface of O-ring tested in 90% CO_2 in methane, crack direction propagation was derived from the convergence direction of the riverlines.

Fig. 2 displays the mirror, mist and hackle areas in the region A, and the crack direction is indicated by the direction in which the riverlines converge to form hackles. The mirror region is associated with slow crack propagation, and the boundary of the mirror region marks the transition of crack speed from a slow and stable speed to rapid acceleration. Hackle lines are formed as a result of smaller cracks branching into one crack. Hackle regions tend to appear in regions where the stress field is changing rapidly either in direction or magnitude [6].

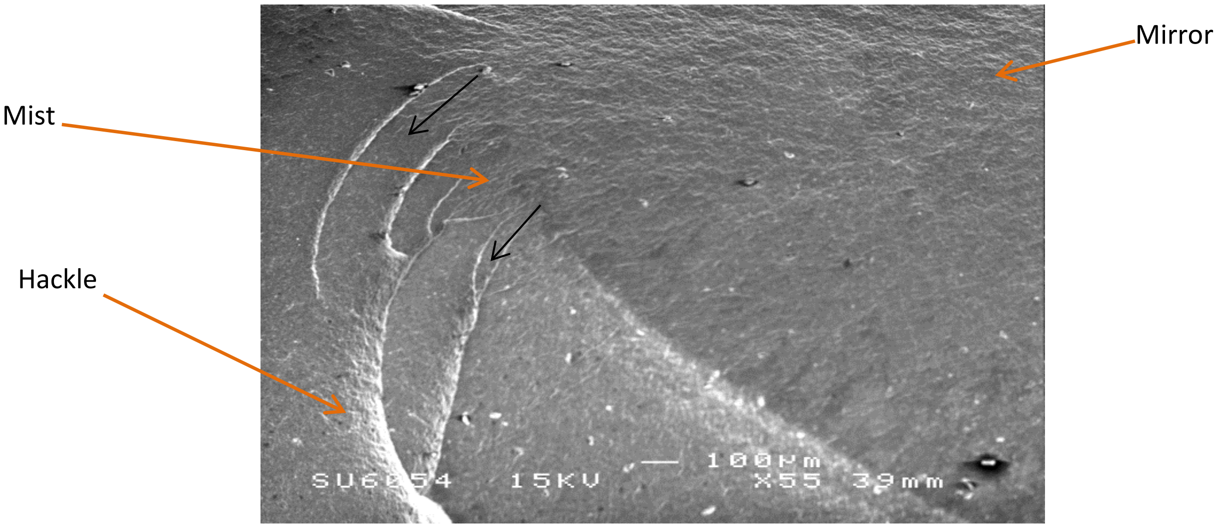

Fig.2 Mirror, mist and hackle morphology of the fractured surface of O-ring tested in 90% CO_2 in methane, the crack direction is indicated by the arrows.

Fig. 3 is a magnification of the mirror region in Fig. 2. The fracture surface displays a dimpled fracture surface containing pits. N. M. Mathew and S. K. De [7] conducted tearing tests on natural rubber reinforced with clay particles and they observed a rough fracture surface containing pits/cavities similar to the fracture surface displayed in Fig. 3. They postulated that the formation of these pits on the surface was a result of reinforcing agglomerates, coming out of the matrix and these loose agglomerates in the matrix act as stress raisers and offer an easy path for the tear to follow, thereby reducing the overall strength of the material.

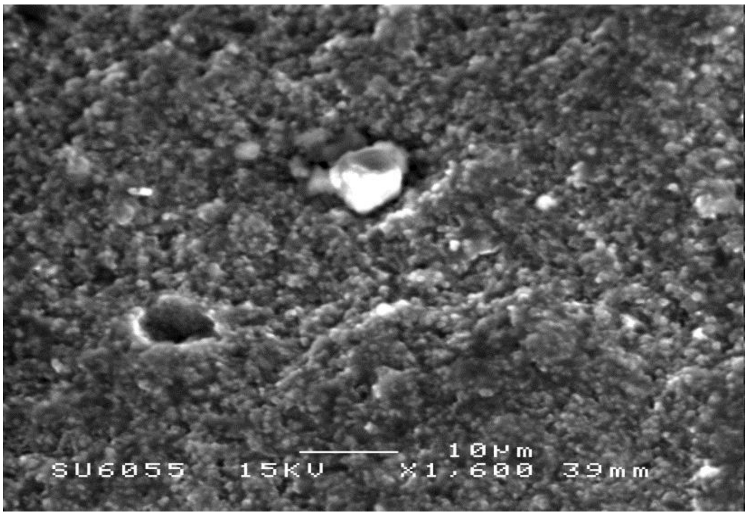

Fig.3 SEM image showing mirror region of the fractured surface of O-ring tested in 90% CO_2 in methane, showing formation of pits on the fracture surface.

The fracture surface of the second O-ring tested under 90% CO_2 in methane also contains two distinct regions similar to the previous O-ring. In Fig. 4 stable crack growth is more pronounced on the inner circumference of the O-ring whilst the outer region displays a rapid crack propagation fracture surface. The direction of the converging hackles indicate that cracks initiated in the centre region and propagated towards the inner circumference of the O-ring and once the cracks reached a critical size, the crack accelerated resulting in rapid crack propagation fracture.

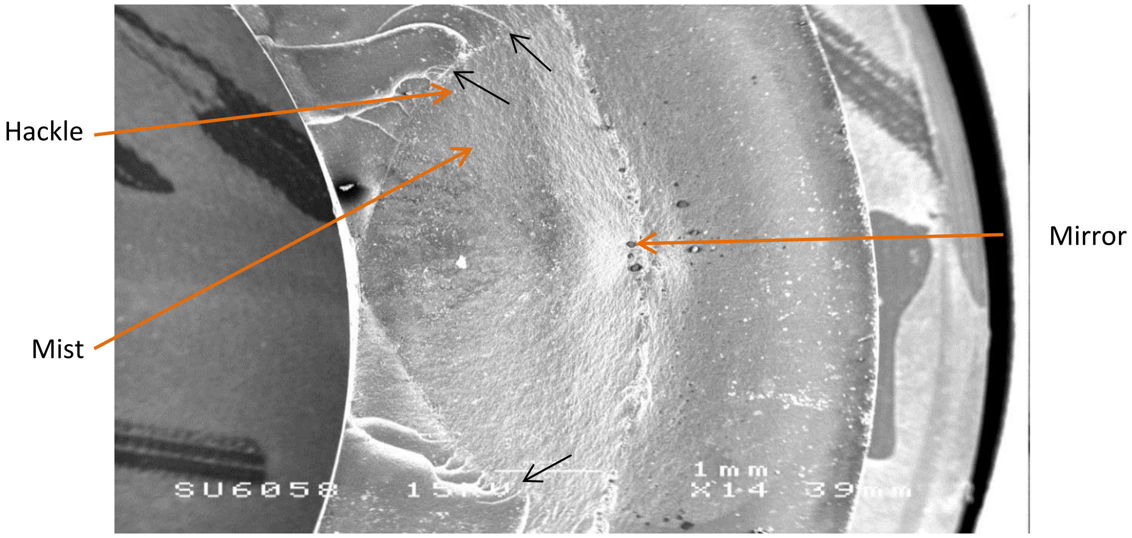

Fig.4 Mirror, mist and hackle morphology of the fractured surface of O-ring tested under 90% CO_2 in methane, the crack direction is indicated by the arrows.

Fig. 5 shows the centre region of the fractured surface shown in Fig.4. Cracking was more pronounced in this region and this is an indication that either higher stress levels where experienced in this region of the O-ring during R.G.D. or this region is a point of weakness in the O-ring structure. The fracture surface in Fig. 5 shows cracks propagating from cavities and these open cavities were a result of inherent voids present in the material rupturing during rapid gas decompression. These cavities act as crack initiation sites in the O-ring thereby reducing the materials strength.

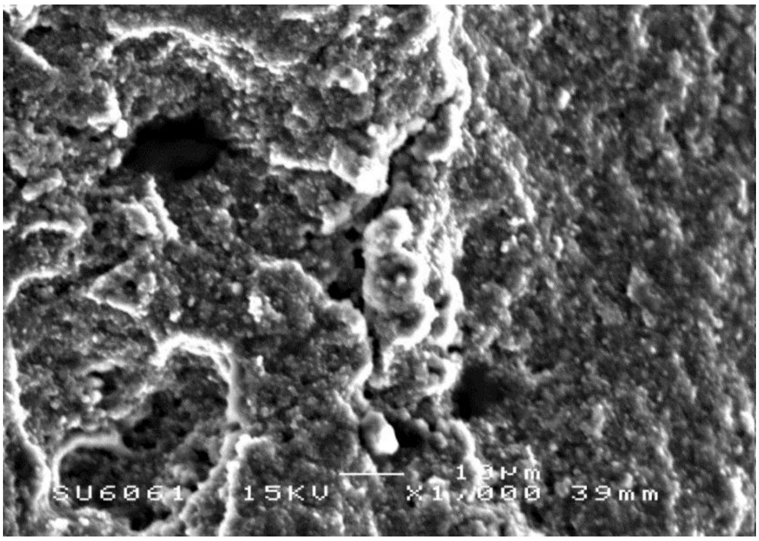

Fig.5 SEM image of the centre region in O-ring tested under 90% CO_2 in methane, displaying cracks propagating from cavities.

Fig. 6 displays a dimpled fracture morphology containing multiple tearing lines, which has been observed on the fracture surfaces of previous specimens. A dimpled fracture surface in rubber is reported to be a result of fracture under a combination of tension and compression loadings, Anil K. Bhowmick [8]. Dimples indicate debonding at the particle/matrix interface and result from a crack passing through debonded particles, A. K. Bhowmick et al [9].

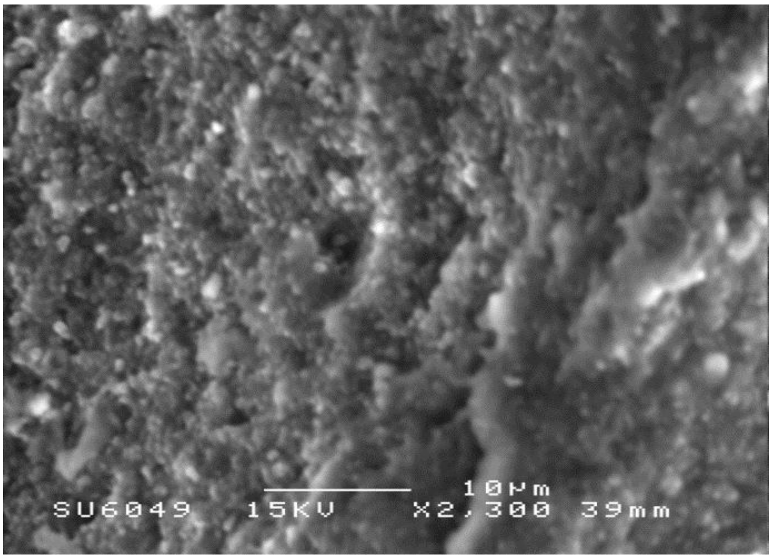

Fig.6 Crack initiation region in O-ring tested in 70% CO_2 in methane, displaying a dimpled fracture surface

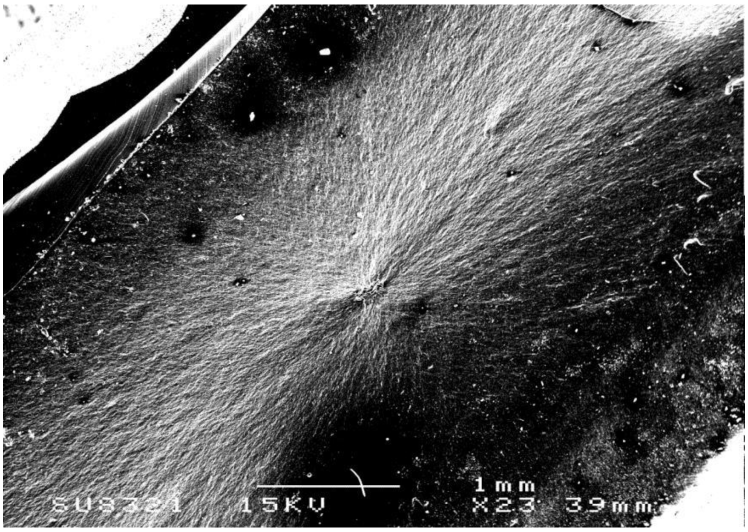

Fig.7 Fracture surface of a spring seal displaying multiple small cracks initiating from an inherent flaw.

Fig. 7 displays the fracture surface of a spring seal containing small multiple cracks propagating from an initiation point in the centre of the spring seal. The relatively rough and bright region in Fig. 7 is the slow propagation region, which is mainly characterised by small multiple cracks propagating away from an initiation point. Similar to O-rings, the spring seals have a higher slow crack propagation region towards the inner circumference compared to the outer circumference. Fig. 8 displays a magnified image of an inherent flaw with cracks propagating at 90^o angle from the flaw. The flaw region contains multiple irregular shaped cavities of different sizes. This type of flaw was likely a result of multiple voids in close proximity rupturing during the decompression cycle and coalescing to form bigger wider voids.

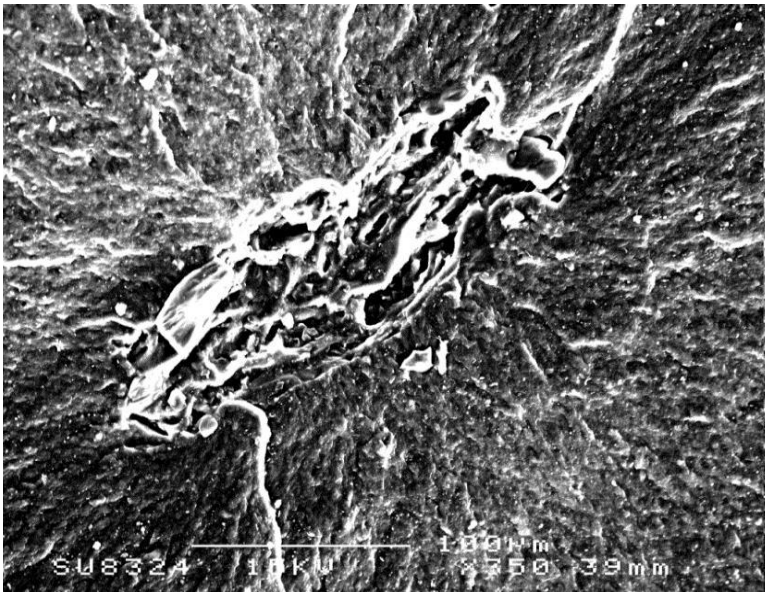

Fig.8 Magnified fracture surface showing microcracks propagating from an inherent flaw.

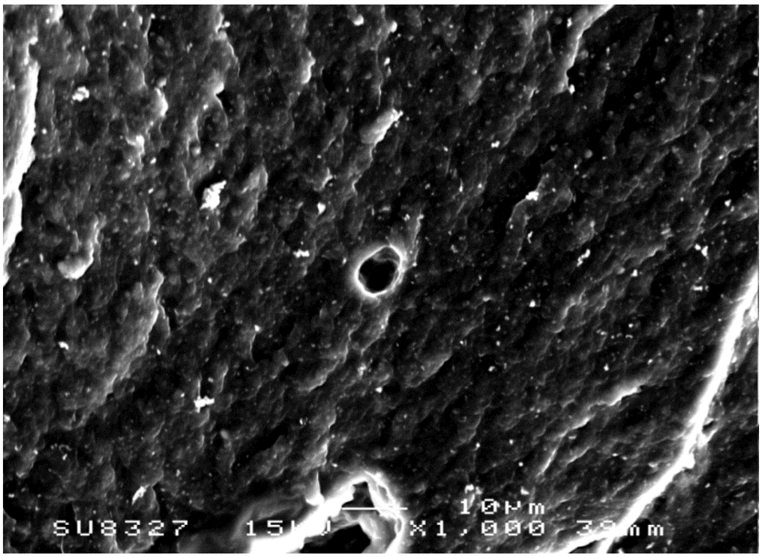

Fig.9 Fracture surface of a Spring seal displaying tear lines and a void present on the fracture surface.

Fig. 9 shows the fracture surface of the spring seal in the slow crack propagation region, the surface contains tearing lines and an open void with a diameter of approximately 7.14 nm. Voids are usually introduced into O-rings and spring seals during processing and are almost impossible to avoid. During rapid gas decompression the voids inflate resulting in tensile stresses or strains in the void walls. If the stresses are higher than the strength of the elastomer, cracks initiate and propagate. The void in Fig. 9 seems not to be the cause for crack initiation in the spring seal, however these open voids in the matrix act as stress raisers and offer an easy path for the tear to follow, thereby reducing the overall strength of the material. Fig. 10 is the fracture surface of a spring seal in the rapid crack propagation fracture region. These regions are characterised by relatively little or no macroscopic visible plastic deformation and they require relatively less energy to form. The fracture surface in Fig. 10 contains pits and particles with varying diameter sizes between 0.736 Nano meters to 9.6 Nano meters.

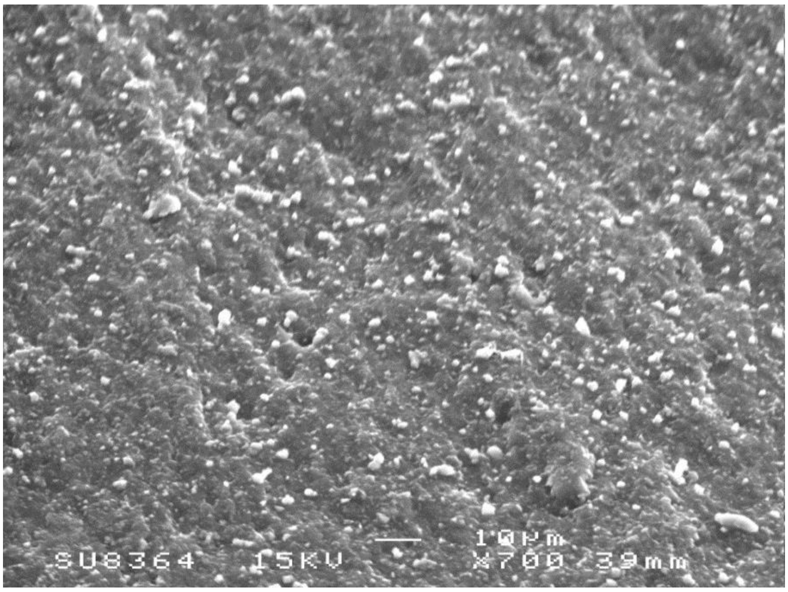

Fig.10 Fracture surface of a spring seal with surface debris and pits.

4. Conclusion

Scanning electron microscopy was conducted on fractured Fluorocarbon elastomer O-rings and Hydrogenated Nitrile Butadiene Spring seals after being exposed to rapid gas decompression. All fractured surfaces contained two distinct regions; rough region which is associated with slow crack propagation, and a smooth region which is normally associated with fast brittle crack propagation, however, rarely associated with elastomers. All three fractured surfaces displayed a dimpled fracture surface in the rough region and this dimpled rough region has been observed by A.K. Bhowmick et al [10] in nitrile rubber, after being subjected to flexural load until fracture. In this experiment nitrile rubber was subjected to repeated tension and compression loadings. They also observed a similar dimpled fracture surface with tearing lines in tear fractures of natural rubber. N. M. Mathew et al reported in their experiments that repeated tension and compression loading generated dimples on the fracture surface. With an understanding of the loading conditions of O-rings in service and after analysing the fracture surfaces, it is possible to conclude that these O-rings failed under tension and compression loadings. Microcracks initiated from microvoids or inherent flaws in the centre region of the O-ring and propagated towards the inner and outer circumference of the O-ring. When the crack size reached a critical size, the crack rapidly accelerated resulting in a catastrophic fracture.

All the flaws where located in the centre region of the spring seals and this indicates that these flaws must have been introduced during the manufacturing process and were not necessarily inherent flaws contained in the elastomer raw material. All fracture surfaces for both O-rings and spring seals displayed more pronounced relatively stable crack growth towards the inner circumference. This indicated that higher stress levels were experienced on the inner circumference of the specimen during rapid gas decompression, or this region may be a point of weakness in both the O-ring and spring seals structures. The fracture surface of both O-rings and spring seals specimens contained pits on the surface and these pits were a result of reinforcing agglomerates, coming out of the matrix. These loose agglomerates in the matrix acted as stress raisers also offering an easy path for a tear to follow thereby reducing the overall strength of the material.

5. References

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