

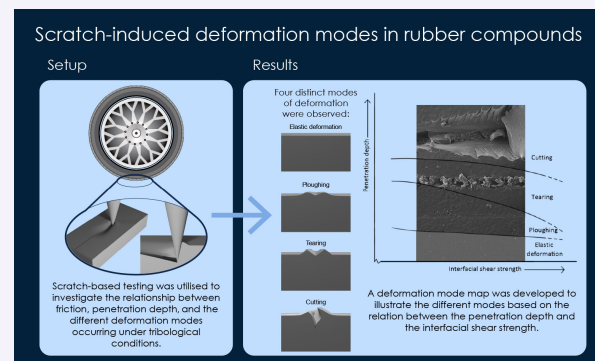
Scratch-induced deformation modes in rubber compounds

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ABSTRACT: Transitions in tribologically induced deformation and wear are well established for metals and polymers, through scratch-based deformation maps, yet a comparable framework for elastomeric materials is still lacking. In particular, the relationship between frictional response and deformation mode transitions in rubber remains insufficiently established. In this study, single-asperity scratch-based testing was employed to systematically investigate the relationship between friction, penetration depth, and deformation behaviour in four elastomer compounds under dry and lubricated conditions. Four distinct deformation modes, namely elastic deformation, ploughing, tearing, and cutting were identified and linked, not only to visual damage but also to characteristic frictional response signatures under both constant and increasing penetration depth. Scratch-based tests were conducted in dry and lubricated conditions. Elastic deformation is characterised by low friction and a small deviation in the amplitude of the frictional force along a single scratch. For ploughing, higher friction is measured along a slightly higher deviation of the amplitude of the frictional force. The tearing mode shows high friction, though, the amplitude of the frictional force over the scratch length still remains low. The cutting mode exhibits both a high frictional force and large amplitude variations in this signal, primarily due to the stick slip phenomena, as commonly known for rubber materials. Lubricating the samples decreased the coefficient of friction and increased the penetration depth required to generate a transition in deformation mode of the material. A deformation mode map was developed to illustrate the different modes based on the relation between the penetration depth and the interfacial shear strength. This deformation map contributes to a better understanding of rubber deformation under varying tribological circumstances. By capturing the initial deformation behaviour, the deformation map could provide valuable insight into the mechanisms driving wear evolution.

KEYWORDS: rubber wear; abrasive wear; deformation characteristics; friction; scratch testing



1 Introduction

Elastomeric materials are subjected to complex tribological loading conditions in a wide range of engineering applications. Under sliding and abrasive contact, local deformation may result in surface damage, crack initiation and, ultimately, material loss. Understanding the mechanisms governing these transitions is therefore important for improving the durability and wear resistance of rubber components.

Tyre tread compounds provide a particularly relevant example, as their interaction with road asperities gives rise to repeated deformation and damage processes. Depending on operating conditions, different wear mechanisms may become dominant; abrasive wear, including cut-and-chip wear, fatigue wear, and

adhesive wear [1]. These mechanisms are influenced by a combination of material properties, contact conditions, and environmental factors. For passenger-car tyres, abrasive and fatigue wear are generally considered the dominant wear mechanisms, while cut-and-chip wear becomes increasingly important in more severe applications such as truck and off-road tyres [2–4]. Localised interactions with sharp asperities can induce substantial deformation and damage in the rubber surface before actual material removal occurs. Characterising these early-stage deformation processes may therefore provide valuable insight into the mechanisms governing wear development in elastomeric materials.

One method to investigate a material's friction and deformation behaviour is by scratch based testing. Xu et al. [5]

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described three laboratory testing methods to study rubber wear: sliding with a needle (scratch-based testing), sliding of a blade, and impact of hard particles. Scratch-based testing of elastomeric materials has been explored by several researchers. Fukahori et al. [6] investigated the surface characteristics of rubber to better understand the features formed during scratching. This work demonstrated the stick-slip behaviour and showed correlations between normal load and both the spacing between the cracks and the coefficient of friction, using varying indenter angles. Low and Teo [7] characterised the influence of carbon black on the scratch and deformation behaviour of styrene butadiene rubber (SBR), neoprene, and ethylene propylene diene monomer (EPDM) compounds using pendulum-based scratch testing. They concluded that the scratch resistance increased for SBR and neoprene compounds with higher carbon black content, while the scratch resistance decreased with increasing load.

Schallamach [8] examined filled and unfilled rubber compounds by introducing damage with a needle and found that different levels of damage and friction occurred depending on the compound formulation. Zhu et al. [9] investigated the scratch behaviour of hydrogenated nitrile butadiene rubber (HNBR) as a function of thermal aging. Their study reports that different types of tearing occurred and that the scratch resistance is significantly influenced by thermal aging. Maeda et al. [10, 11] scratched NR, BR, and SBR with a pendulum scratching test setup defining a relation between the frictional force and the observed damage. Their study showed that the $\tan\delta$ might be the dominant factor influencing the friction generated during the scratch-based tests. However, they concluded that the friction generated is dependent on both its deformational and adhesive term. These publications provided initial insights into scratch-based testing of elastomeric materials. However, as shown by Xu et al. [5], little is known about rubber behaviour within scratch-based testing and the definition and characterisation of different deformation modes under varying conditions for rubber materials has not been addressed in literature yet.

In contrast to elastomers, deformation modes and wear maps have been developed for metals and polymers to visualise the conditions under which specific types of damage occur. Scratch-based testing of polymers has been widely explored. Brostow et al. [12] investigated a method to assess wear and elastic recovery after scratching in polypropylene, polyester, and Teflon under different forces and increasing numbers of scratches along a single groove. Iqbal et al. [13] constructed scratch deformation maps under various contact conditions for polyether ether ketone (PEEK) surfaces, represented in graphs based on lubrication, normal load, scratch depth, temperature, and nominal contact strain. An et al. [14] evaluated the scratch resistance of polymethyl methacrylate (PMMA) and identified three damage modes, defined as ploughing, whitening, and cutting.

Hokkirigawa and Kato [15] developed a wear map and concluded that abrasive wear of metals occurs in three stages: ploughing, wedge or prow formation, and cutting. These stages of wear were defined by scratching several metals with a conical indenter with a spherical tip under both dry and lubricated conditions, and are mapped into a transition diagram. The axes of this diagram were defined as interfacial shear strength and degree of penetration. The interfacial shear strength is dependent on the degree of the friction influenced by lubrication and the degree of penetration depends on the hardness of the material, the geometry of the indenting tip, and the applied load. Hokkirigawa and Kato [15] based their results on equations derived by Challen and Oxley [16] who defined slip line models and derived equations for soft

asperities deformed by hard asperities. With these equations, the interfacial shear strengths were defined.

It can therefore be concluded that literature has shown that scratch-based testing can be a powerful tool for characterising deformation and wear modes within metals and polymers. The research on scratch-based testing for metals and polymers can be utilised as a basis to extend the research in scratch-based testing for elastomeric materials, as a fundamental study to generate new insights in rubber friction and deformation. In this work, four rubber compounds were scratched with a single asperity indenter under varying contact conditions, aiming to identify deformation mode transitions in elastomeric materials and to establish a deformation-mode framework that relates contact conditions, frictional response, and observed damage behaviour. The purpose of this study is thus twofold. Firstly, to explore whether the expected transitions in rubber damage can be distinguished by varying test conditions in a single asperity ploughing test and secondly, to develop a deformation diagram based on the observations and results utilising scratch-based testing.

2 Materials and experimental setup

2.1 Compound formulation and preparation

Four rubber compounds were used for this study, these compounds are designed to investigate a broad variety of rubber matrices and reinforcing fillers, typically used in passenger car and truck tyre tread compounds [17–19]. Natural rubber (NR, SMR-10) and styrene butadiene rubber (S-SBR Sprintan SLR 4602, Synthos Schkopau GmbH) in combination with butadiene rubber (BR, Buna CB24, Arlanxco Deutschland GmbH) filled with carbon black N330 (CB, Corax N330, Orion Engineered Carbons GmbH) and silica ULTRASIL 7000 GR (Evonik Industries, Wesseling, Germany) were mixed. Bis(triethoxysilylpropyl)tetrasulfide (TESPT, Si69, Evonik Industries, Wesseling, Germany) was used as coupling agent and sulphur (α -Sulphur, Eastman Chemical Corporation) as curative. N-cyclohexyl-2-benzothiazole sulfenamide (CBS, Flexys, Belgium) was used as primary accelerator and diphenyl guanidine (DPG, Rhenogran DPG, Lanxess Deutschland GmbH) as secondary accelerator. Treated distillate aromatic extract (TDAE, Vivatex 500, Hansen and Rosenthal KG, Hamburg, Germany) was added as processing oil, zinc oxide (ZnO, ZnO RS RAL 844 C, Arnspurger Chemikalien GmbH) and stearic acid (Edenor ST1 GS, Emery Oleochemicals, Düsseldorf, Germany) as activators, with paraffin wax (Protektor G 3108, Paramelt B.V.) and N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD, vulkanox 4020/LG, Lanxess Deutschland GmbH). The exact compound formulation of the four compounds used in this work can be found in Table 1.

All compounds were mixed in three stages, in an internal mixer Brabender Plasti-Corder 350S (Duisburg, Germany). These stages include an initial mixing stage, salinization stage for optimal salinization, and a third stage for addition of the curing package. The compounds were mixed on a two roll mill after each stage in the internal mixer. The samples have been cured in a hydraulic press (Wickert Maschinenbau GmbH, Germany) according to their t_{90} at 150 °C, measured with an RPA TA Elite (TA Instruments, New Castle, Delaware).

A polished tool steel WN 1.2379 conical tip with a 30° cone, a 242 HV1 hardness, and 0.11 μm Ra surface roughness was utilised as the scratching indenter. A 30° cone was utilised as it enabled accurate identification of the deformation modes in rubber within the chosen penetration-depth range.

Table 1 Compound formulation for four selected compounds (unit: parts per hundred rubber)

Ingredient	NR-CB60	NR-S60	SBR-CB60	SBR-S60
NR	75	75	0	0
SBR	0	0	75	75
BR	25	25	25	25
CB N330	60	0	60	0
Ultrasil 7000 GR	0	60	0	60
Si 69	0	4.8	0	4.8
TDAE	10	10	10	10
ZnO	3	3	3	3
Stearic acid	2	2	2	2
Wax	1.5	1.5	1.5	1.5
6PPD	2	2	2	2
DPG	0.3	1.2	0.3	1.2
CBS	1.5	1.5	1.5	1.5
Sulfur	2.4	1.8	2.4	1.8

Note: The DPG and sulphur amount were adjusted according to the filler system.

2.2 Experimental setup

A universal mechanical tester (UMT, Bruker, USA) equipped with a 60 N loadcell and programmable moving stages in x -, y -, and z -direction was used to conduct the single asperity scratch-based experiments. Figure 1 provides a visual representation of the experimental setup.

2.3 Test conditions

All experiments were performed in depth-control mode. Two testing series were undertaken. The first test was conducted by linearly increasing the penetration depth and secondly a series of tests was conducted at constant depths for each material in both dry and lubricated conditions. Lubrication was used to characterise the influence of the adhesion part of friction. In the presence of a lubricant, the adhesion is minimised. A paraffinic white oil, Finavestan A 360B (TotalEnergies, France), was used as

lubricant. For both test series, the indenter initially identified the surface to ensure that the input depth and penetration depth in the sample were the same. In the first test, the indenter moved in x -direction while the penetration depth linearly increased. In the second test, after the surface was identified, the input depth was slowly reached. Hereafter, the indenter scratched with a constant penetration depth. Following each test, the indenter was lifted and returned back to the zero position. Hereafter, the indenter was moved in y -direction, for a clean surface for the next scratch.

For the first test, the scratch length was set at 30 mm with the penetration depth ranging from 0 to 1.2 mm. The penetration velocity was 0.04 mm/s and the scratch velocity was 1 mm/s. The test specimens had a diameter of 52 mm and a height of 12 mm. For the second test, the scratch length was 15 mm. The test specimens used, had a diameter of 28 mm and a height of 12 mm. The scratch depth was set in a range of 0.1–1.1 mm in steps of 0.1 mm. All four compounds were scratched over this range under dry and lubricated conditions. Tests were repeated a minimum of three times to assure repeatability and an average curve of each test condition was selected.

2.4 Data analysis

The data were analysed and processed using different methods. The output data generated by the UMT, are the frictional force (F_f), the normal load (F_n), the coefficient of friction (μ), the initial penetration depth, and the penetration depth along the scratch length. MATLAB was used to analyse these data. For the second test series, average values were generated over the distance between 5 and 12 mm of the scratch length, which was selected as a stable region for analysis.

To visually analyse the samples and define the residual depth remaining after scratching, different techniques were used, including a KEYENCE VHX-7000 digital microscope (KEYENCE, Osaka, Japan), a Sneox confocal microscope (Sensofar-tech, Terrassa, Spain), and a scanning electron microscope (SEM) (Phenom XL, Thermo Fisher Scientific, Phenom XL, Waltham, MA, USA). The results from these techniques were combined and used to describe the rubber scratch behaviour and define the transitions between the different deformation modes.

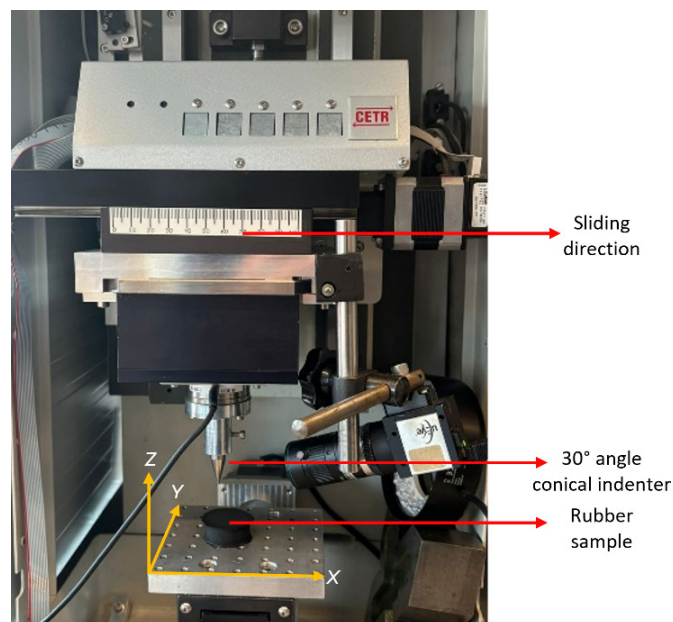


Fig. 1 Test setup of the UMT CETR with moving x , y , and z stages, a 30° angle conical indenter, and a rubber test sample.

3 Results and discussion

In order to determine the feasibility of applying scratch testing to determine wear mode transitions in rubber compounds, firstly, a focus was given to NR-CB60, to discuss the numerical data and the SEM and microscope images in more detail. All results for this compound will be presented and discussed elaborately, whereafter, the results of all other compounds will be discussed and compared. The results of the initial scratch tests for increasing penetration depths are presented in Fig. 2.

Within these experiments, the indenter scratches with a linearly increasing penetration depth along the scratch length. The results show the visual (Fig. 2(a)) and frictional (Fig. 2(b)) responses. Figure 2(a) shows the different deformation modes along the scratch length as the severity of the deformation increases. Four deformation modes can be distinguished from Fig. 2(a): elastic deformation, ploughing, tearing, and cutting. Elastic deformation is defined as the region of scratched materials which shows a fully recovered surface of which the height of the recovered track is within the surface roughness of 2 μm . The ploughing mode consists of permanent deformation. In this mode, a depression greater than the surface roughness is discernible and material can be seen to have been permanently displaced to the sides of the track. The third mode, termed tearing, is characterised by the initiation of surface cracks and a low degree of tearing in and around the scratch track, though, a significant degree of elastic deformation and ploughing can also be observed. In the last mode, termed cutting, the indenter cuts through the rubber and effectively signals full material failure. A schematic representation of the cross-sectional profiles of each deformation mode is presented in Fig. 3. These profiles are based on optical microscopic, SEM, and confocal measurements.

Although similar observations in rubber deformation behaviour under scratching were made, as previously published by Refs. [8–11], whose studies are further elaborated upon in the introduction, these results show that scratch-based testing is a suitable method to indicate clear transitions in material damage ranging from elastic deformation to full material failure.

Comparing dry and lubricated results, Fig. 2(a) shows later transitions for each mode, at deeper penetration depths, for the

lubricated samples compared to the dry condition. Figure 2(b) shows the corresponding frictional force output data. It shows that the frictional force for dry condition is higher than for the lubricated condition, an observation that can be expected due to the drastic decrease of adhesive friction as also stated by Moore and Geyer [20] and Lorenz et al. [21]. The observation can also aid in explaining the later transitions in deformation modes in the lubricated condition compared to the dry condition, as the rubber damage seems to be dependent on the generated friction, as stated by Maeda et al. [10]. Furthermore, it can be observed that the amplitude of the signal increases with increasing penetration depth. Considering Figs. 2(a) and 2(b) together, it can be seen that in the first two stages of deformation, elastic deformation and ploughing, the amplitude of the signal is low, it increases for tearing and increases further for cutting. The fluctuations in this signal relate to stick slip phenomena, which has also been described by Low and Teo [7], who conclude that the increase in normal load intensifies the stick-slip pattern.

To further investigate the damage behaviour in more detail and to separately investigate each deformation mode, a series of scratches performed at constant penetration depths per scratch was conducted. NR-CB60 was scratched in dry and lubricated conditions for penetration depths ranging between 0.1 and 1.1 mm. Characteristic results are visualised by a scanning electron microscope and are represented in Fig. 4.

The SEM images in Fig. 4 illustrate the different deformation modes within rubber damage which were also identified in Fig. 2(a). Figure 4(a) shows elastic deformation, as no residual scratch is visible. For Fig. 4(b), the damage relates to the beginning of mode three, tearing, as it shows initiations of crack formation. These superficial cracks are better visible in Fig. 4(c) in which the residual scratch of the 0.6 mm penetration depth shows further tearing. Scratching in dry condition with a 0.8 mm penetration depth shows the fourth deformation mode, cutting. Scratching with a 1.0 mm penetration depth shows more severe damage, though, the behaviour is still similar to the 0.8 mm scratch. In Figs. 4(d) and 4(e), the cutting behaviour is represented by the deep residual scratch implying failure of the material.

In addition, Figs. 4(f)–4(j) show the results of scratching in lubricated condition. It can be seen that the transitions occur for a deeper penetration depth as is in line with Fig. 2(a). There are noticeable differences in the transitions between the dry and lubricated condition. In Fig. 4(b), at a penetration depth of 0.4 mm in dry condition, the deformation mode can be classified as tearing, as observed by the small superficial cracks. In Fig. 4(g), in the lubricated condition for the same penetration depth, no tearing is noted as the deformation mode here is ploughing, as

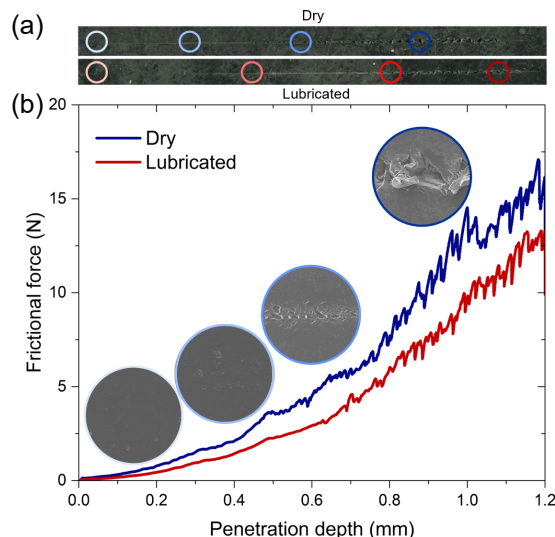


Fig. 2 (a) Visual representation of linear increasing penetration depth scratches and (b) the associated frictional force data, for dry (blue) and lubricated (red) conditions for NR-CB60 including images of the four deformation modes in dry condition.

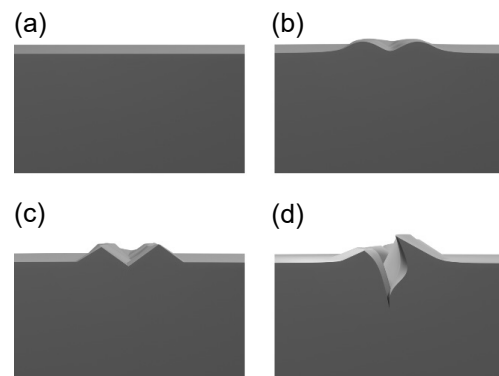


Fig. 3 Schematic representation of the cross-sectional profiles of (a) elastic deformation, (b) ploughing, (c) tearing, and (d) cutting.

characterised by the residual deformation and material which is moved to the sides. This is also visible for Fig. 4(h) with a penetration depth of 0.6 mm. When the penetration depth increases further to 0.8 mm in dry condition, severe cutting is observed, resulting in failure of the material. The tearing deformation mode is only visible in lubricated condition for the 0.8 mm penetration depth and cutting and failure of the material are visible from depths of 1 mm. These results clearly indicate large differences in scratch behaviour for dry and lubricated

conditions. The differences in deformation modes seen in Fig. 4, can also be linked to distinct features in the frictional force response data as shown in Figs. 5(a) and 5(b) for dry and lubricated conditions respectively.

The graphs show the frictional force along the scratch length for each penetration depth represented in Fig. 4. It shows that in lubricated conditions lower frictional forces are generated due to the decrease in adhesive friction. Figure 5(a) shows a larger amplitude for the signals of 0.8 and 1.0 mm penetration depth

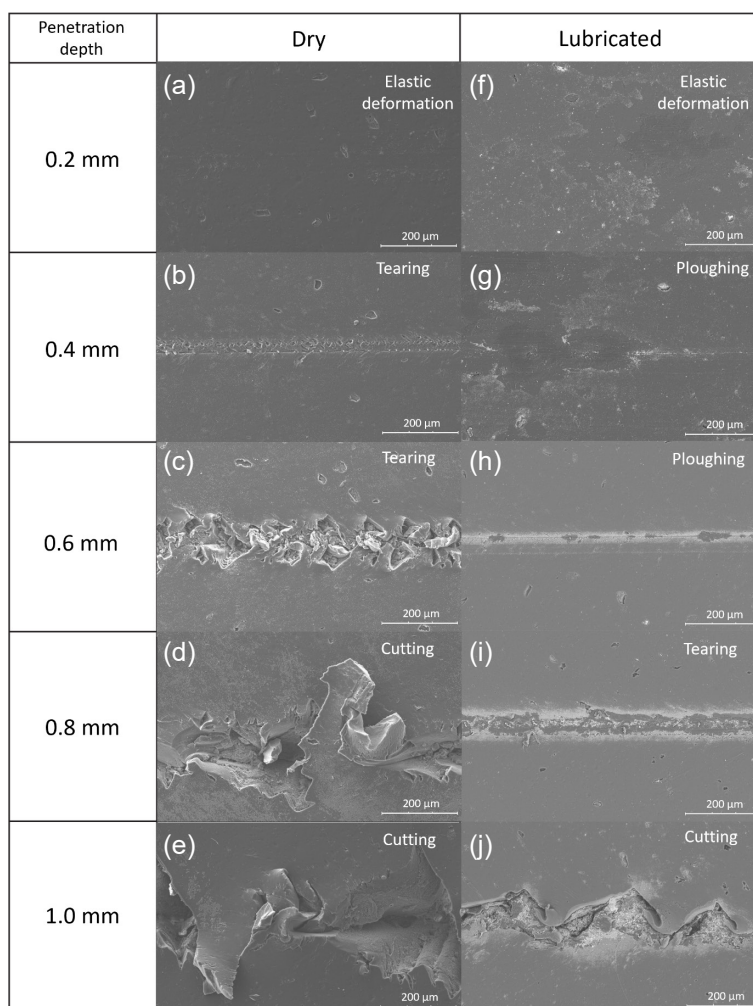


Fig. 4 SEM images of NR-CB60 of 0.2, 0.4, 0.6, 0.8, and 1.0 mm penetration depth for dry (a–e) and lubricated (f–j) conditions with a 30° cone angle.

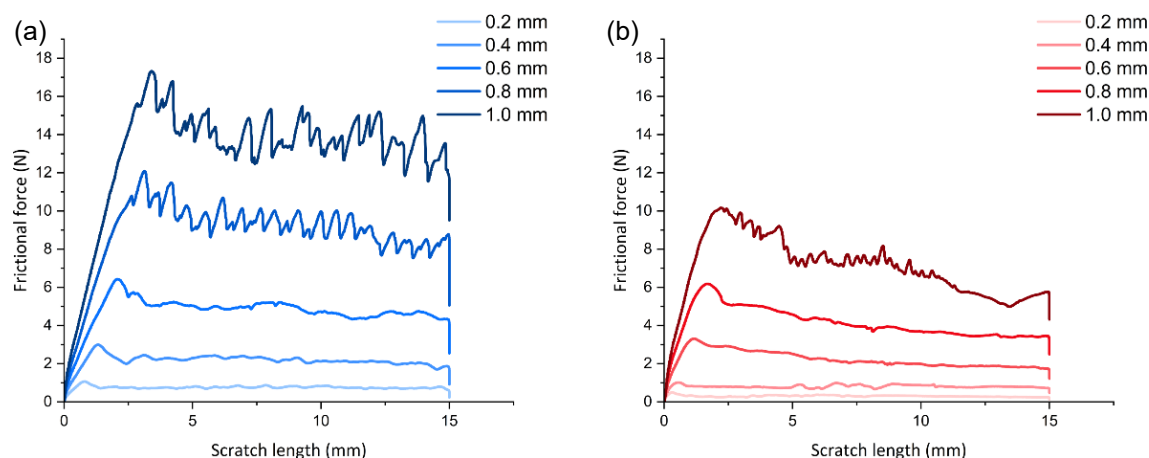


Fig. 5 Frictional force data of NR-CB60 along the scratch length for varying penetration depths in (a) dry and (b) lubricated conditions.

compared to 0.4 and 0.6 mm penetration depth, indicating cutting and failure of the material as the larger stick-slip response arises, as was confirmed by the visualisations of Fig. 4. The transition of the tearing to the cutting regime can therefore be effectively characterised by the change in amplitude of the frictional force signal along the sliding distance.

For the lubricated condition, Fig. 5(b) clearly shows larger variations in amplitude for the 1.0 mm penetration depth in comparison to the 0.8 mm and less, indicating only failure of the material for 1.0 mm. The dry condition shows a larger amplitude than the lubricated one, comparing the 0.8 and 1.0 mm of the dry condition with the 1.0 mm of the lubricated condition. This larger amplitude indicates a larger stick-slip response and more severe cutting, as is confirmed by Fig. 4. The signal of the 0.8 mm penetration depth in lubricated condition is as smooth as the lower penetration depths, showing no cutting, which is according to the results of Fig. 4. There are no statistical differences in the variation of the amplitude of the frictional force between elastic deformation and ploughing and between ploughing and tearing, because for elastic deformation and ploughing no cracks are initiated and the tearing mode only includes superficial cracks indicating less dominant stick slip behaviour. Thus, no conclusion for identifying elastic deformation, ploughing, and tearing can be drawn based on the amplitude of the frictional force signal.

As can be observed from Fig. 5, the output data from the scratch tests include a ramp-up phase, which is dependent on the target penetration depth, followed by a representative, stable region. To generate comparative values for each test condition, average values, the arithmetic mean, for the frictional force, for the normal force required to maintain the prescribed constant penetration depth, and for the resulting coefficient of friction were created by the MATLAB software for each test in both dry and lubricated conditions, based on the data recorded between 5 and 12 mm scratch length. The results are presented in Fig. 6, including the different deformation modes occurring for each penetration depth, demarked by the symbols.

Figure 6 shows an upswing in the normal load and shows that the dry and lubricated conditions only slightly differ across the full range of testing, since the load needed to reach a certain penetration depth is dependent on material properties and the indentation geometry and not significantly influenced by lubrication. The slightly higher values recorded for the dry contact can be attributed to the higher friction between the indenter and the material during scratching due to the slightly increased local slip in the lubricated condition [22]. Figure 6(b) also shows an upswing trend in the frictional force; however, it shows a large

decrease in frictional force compared to the normal load when scratching in a lubricated contact. It shows that the frictional forces of the lubricated conditions are slightly less than half of the frictional force for the dry condition for all penetration depths. This indicates that, the influence of the lubricant remains constant throughout all penetration depths and remains present in the sliding contact. This relatively constant relation between the dry and lubricated conditions can also be seen in Fig. 6(c), where the coefficient of friction shows a linear relationship with the penetration depth. However, this is in contrast to observations in literature that show that scratching with a conical indenter in other materials, such as metals, would suggest a constant coefficient of friction for low penetration depths or low loads indicating a constant relation between the frictional force and normal load [23, 24]. In the current study case, the linearly increasing coefficient of friction indicates that for increasing penetration depths, the rate of change of the frictional force is greater than that of the normal load. This phenomenon could be explained by the increasing contribution of bulk deformation and the viscoelastic properties of rubber at deeper penetration depths. Figure 6(b) shows that similar frictional forces occur during transitions between deformation modes under both lubricated and dry conditions. This observation indicates that the frictional force can be correlated to the material deformation mode transitions, independent of the lubrication conditions.

3.1 Influence of material characteristics on scratch behaviour

The same characterization that was performed for NR-CB60, was performed for all compounds described in the materials subsection. To further establish the relation between friction and rubber deformation, Fig. 7 shows the relation between the frictional force and the penetration depth for all compounds.

Figure 7 shows that each material has their own trend between the frictional force and penetration depth that becomes more distinguished with higher penetration depths. NR-S60 generates the lowest frictional force for any penetration depth and NR-CB60 has the highest frictional force. However, SBR-CB60 also shows similar high frictional forces. The higher frictional force observed for both carbon black compounds is hypothesised to be most likely due to weaker filler-rubber interactions, which increase the mobility of the system, leading to greater internal friction. These weaker interactions arise from the primarily physical nature of carbon black's interactions with rubber, in contrast to the stronger chemical bonding formed with silica [25].

To draw further conclusions, SEM images of the scratches of all

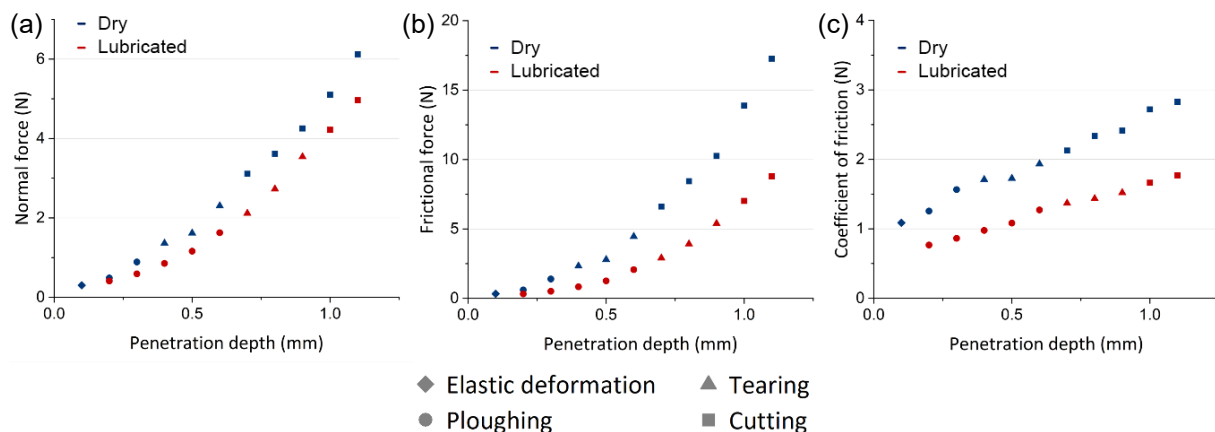


Fig. 6 (a) Normal load, (b) frictional force, and (c) coefficient of friction for varying penetration depths of scratches in NR-CB60 in dry and lubricated conditions.

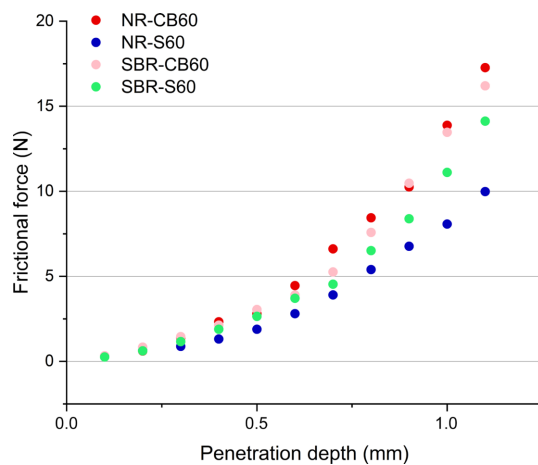


Fig. 7 Correlation between the frictional force and penetration depth for all four materials in dry condition.

four rubber compounds were further investigated. **Figure 8** shows visual representations of the scratch behaviour for dry conditions and **Fig. 9** for lubricated conditions for the same penetration depths as **Fig. 3**.

The images in **Fig. 8** reveal that the carbon black filled compounds, NR-CB60 and SBR-CB60, show similar degrees of damage and that tearing, cutting, and ultimately failure of these compounds occur at lower penetration depths than the silica-filled compounds. Also similarities in scratch behaviour of the polymer types can be observed. The SBR compounds show longer intervals between the stick-slip features than the NR compounds. NR-S60 shows the best scratch resistance, with the deepest penetration

depth needed for failure of the material. It also shows the smallest impacted residual scratch width indicating the most elastic recovery. All other compounds show more comparable results in residual width although, small differences are visible, as is in line with **Fig. 7**.

The visual representations of the scratches under lubrication of **Fig. 9** show a large impact of lubrication on the scratch resistance of the rubbers as the severity of the damage is less compared to the dry conditions at a certain penetration depth. However, it also shows similar trends where NR-S60 shows the best scratch resistance. **Figure 9** also shows similar trends for compounds with the same filler or with the same polymer. For the 1.0 mm penetration depth in **Fig. 9**, the carbon black filled compounds show similar features. The silica filled compounds also appear to exhibit certain characteristic features at a given scratch depth as they show the smallest impacted residual scratch width with the largest elastic recovery. **Figure 8** also shows similarities within the NR- and SBR-based compounds, where the SBR-based compounds show more tearing than the NR-based compounds.

Large differences in the deformation behaviour between the dry and lubricated conditions are visible. This is attributed to the significant decrease in adhesive friction for the lubricated conditions. For the dry conditions, the deformation behaviour is caused by both hysteresis friction and the adhesive part of friction [6, 26, 27], in which for deeper scratches the hysteresis part has a larger impact. Failure of the compounds both in **Figs. 8** and **9** shows periodic cracks, initiated by the stick-slip behaviour of rubber [6]. Damage within the rubber compounds is illustrated in **Figs. 8** and **9**, however no wear, mass loss, could be measured, indicating negligible wear after single scratch tracks. It may be

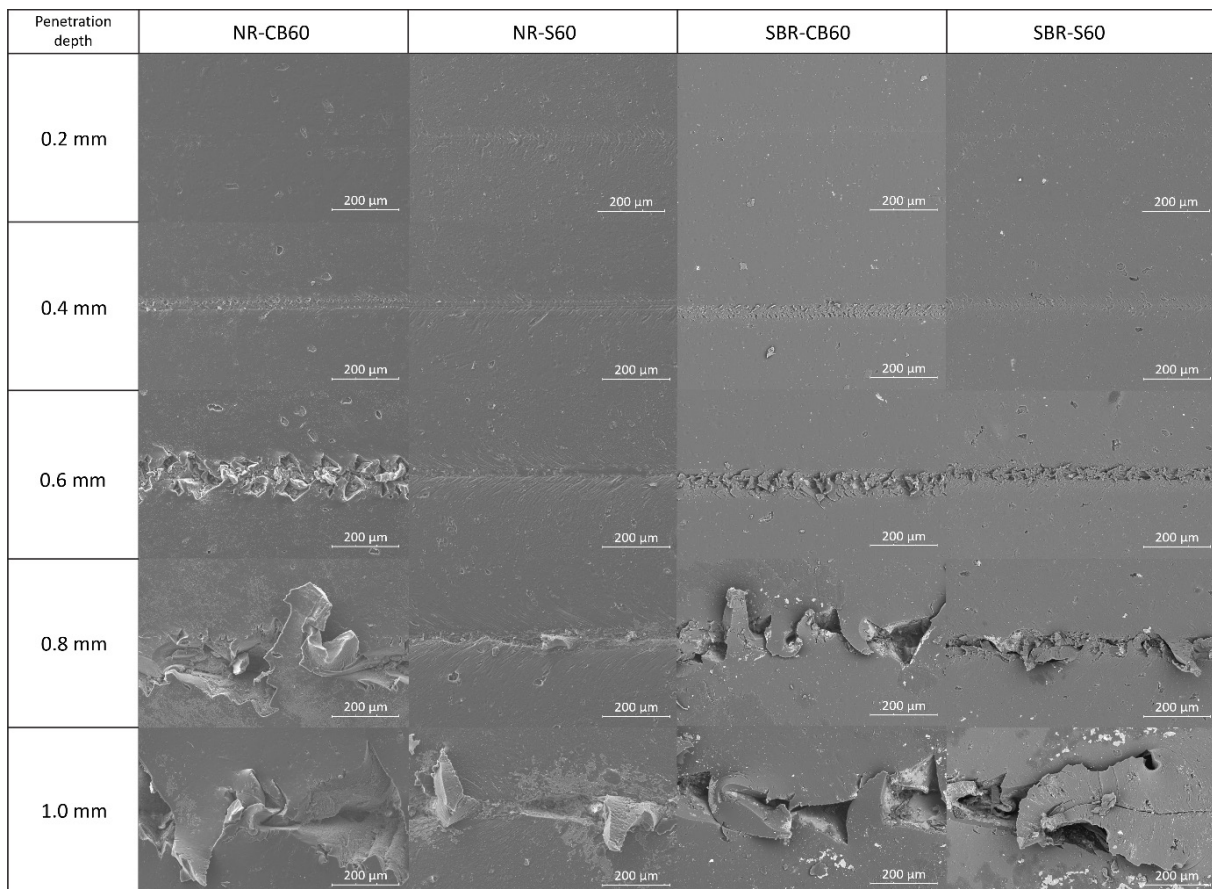


Fig. 8 SEM images of four rubber compounds of 0.2, 0.4, 0.6, 0.8, and 1.0 mm penetration depth for dry conditions.

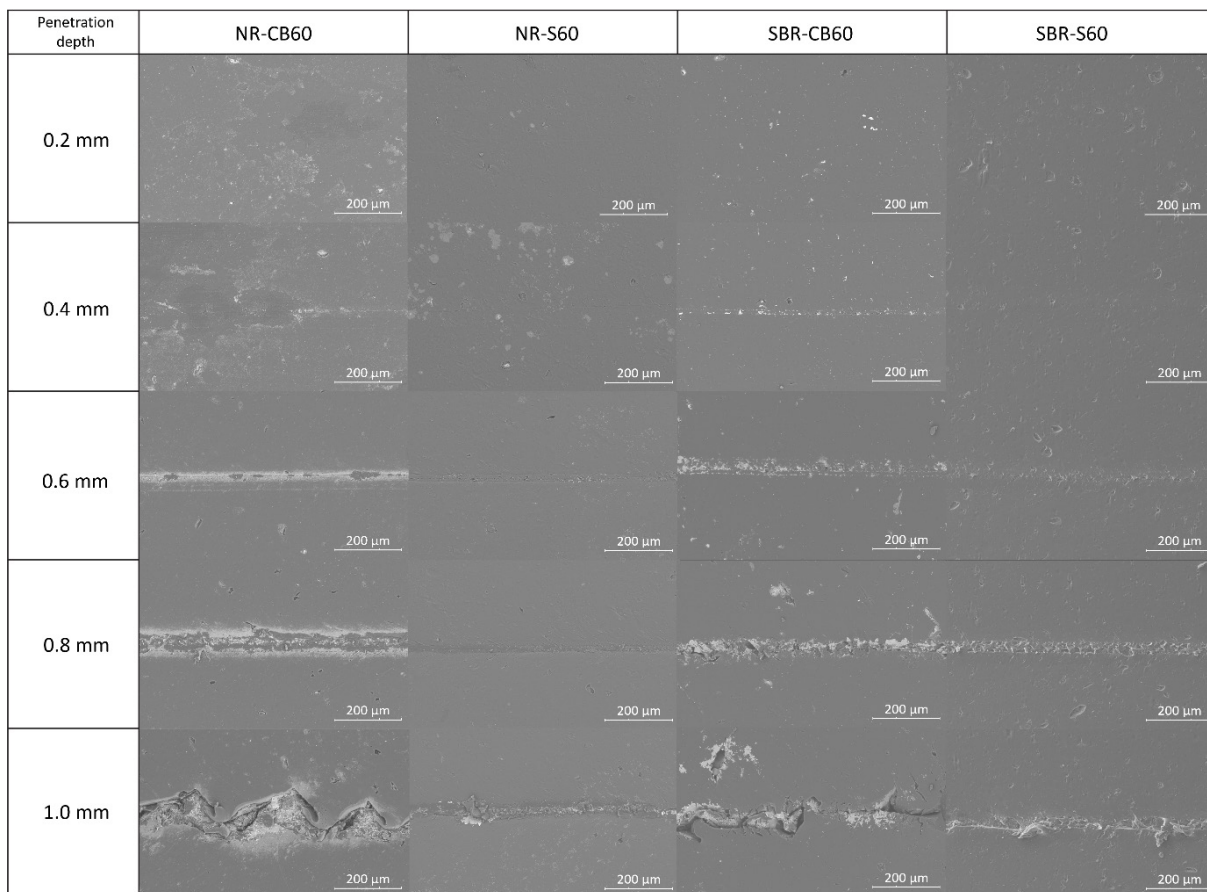


Fig. 9 SEM images of four rubber compounds of 0.2, 0.4, 0.6, 0.8, and 1.0 mm penetration depth for lubricated conditions.

expected that for repetitive scratching, abrasion will be observed, as also stated by Shallamach [8], since these deformation patterns initiated by stick-slip motion are closely related to wear [6, 28].

Correlating Fig. 7 to Figs. 8 and 9, it can be concluded that NR-S60 has the best scratch performance, as a deeper penetration depth is needed for tearing and cutting. Besides, both carbon black filled compounds, NR-CB60 and SBR-CB60, show cutting in Figs. 8 and 9 to occur at the lowest scratch depths and these compounds are also found to reach the highest frictional forces in Fig. 7, indicating a strong relation between the frictional force and the transitions in deformation mode behaviour. Therefore, the filler seems to have more influence on the scratch resistance than the polymer as both carbon black compounds have a lower scratch performance. This can be explained by the dominating effect of an active filler on the reinforcing behaviour of a rubber compound [29]. By combining the SEM and optical microscope images with the numerical data, the four rubber compounds can be ranked from best to worst scratch performance respectively in order of NR-S60, SBR-S60, SBR-CB60, and NR-CB60.

Material characteristics of these four compounds were tested and are shown in Fig. 10. It shows the performance in percentage, where the highest value is taken as 100%. The absolute values are added on top of each column. It demonstrates that the carbon black filled compounds have a higher Shore A hardness than the silica filled compounds in agreement with Mohktari et al. [30]. The higher hardness and higher storage modulus at room temperature, E' RT, can be caused by more filler-filler interactions, that also lead to a higher Payne effect [31], as can be observed in Fig. 10. Besides similarities within fillers, Fig. 10 also shows similarities within the base polymer. The NR-based compounds exhibit a higher tensile strength, mainly due to their

strain induced crystallisation [32]. The NR compounds also show a higher elongation at break compared to SBR, comparing with the same filler, as was also stated by Abdelsalam et al. [33]. As was concluded from Fig. 8, NR-S60 showed the best scratch performance. Ultimately, the performance of NR-S60 might be correlated to the higher elasticity of NR in combination with the better filler-rubber interactions of silica.

The four materials in Fig. 10 are ordered from left to right,

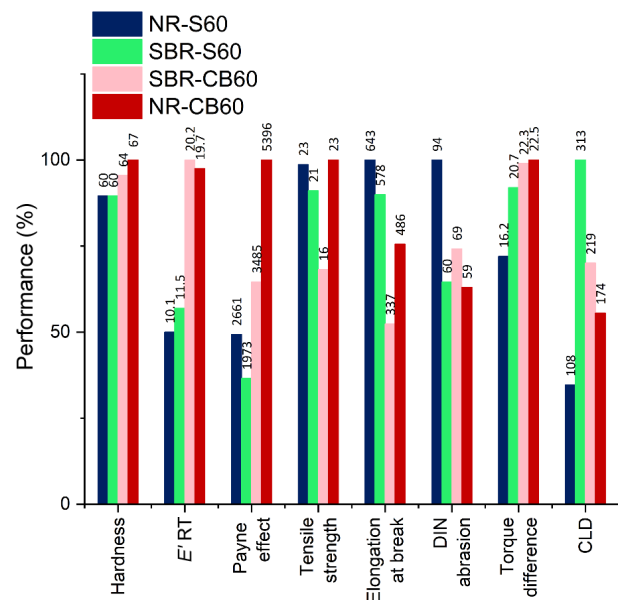


Fig. 10 Material characteristics of the four compounds.

from best to worst scratch performance respectively for each material characteristic. Creating this order will directly show the trend between material characteristics and the scratch performance. Even when only four compounds were compared, some trends are visible. Figure 10 shows the negative correlation between the scratch performance and the DIN abrasion, even if SBR-S60 and NR-CB60 are statistically similar, indicating that materials can have a higher scratch resistance but poor abrasion resistance as was also mentioned by Ozaki et al. [27]. Other trends shown in Fig. 10 are the relationship between the scratch performance and the hardness, torque difference of the cure curve, Payne effect, storage modulus at room temperature taken from dynamic mechanical analysis (DMA), and elongation at break. These relationships indicate that a better scratch resistant material has a lower hardness, lower storage modulus at room temperature, lower Payne effect, and higher elongation at break. However, these results are in contrast with Refs. [8, 9] whose articles stated that an increase in crosslink density and hardness enhances the scratch resistance. Harder compounds would normally have smaller contact area, resulting in lower friction. However, in contrast to previous studies, the penetration depth, in the experiments conducted for this current study, was kept constant with varying normal loads to reach a given depth, therefore the contact area remains (nominally) the same for all compounds for a certain penetration depth. Therefore, a harder compound does not necessarily enhance scratch resistance.

Figure 7 is presenting the frictional force generated during the scratch tests and comparing this with the material properties of Fig. 10. The lower hardness caused by the lower crosslink density of NR-S60 results in the lower frictional force of Fig. 7, due to the lower resistance to deformation induced by ploughing. Additionally, NR-CB60 and SBR-CB60, which generated the highest frictional forces according to Fig. 7, have a higher loss modulus and more hysteresis contributing to a higher deformational friction, as is also stated by Maeda et al. [10].

Another parameter to define the scratch performance is the residual scratch width after testing, as it indicates the degree of elastic recovery. Figures 8 and 9 show a broader residual width for the carbon black compounds mainly due to the type of filler-polymer interactions of carbon black compared to the silica/silane system, which can also be correlated to the higher Payne effect as can be seen in Fig. 10. Another observation from these experiments is that these carbon black filled compounds have the highest hardness and lowest elongation at break, indicating higher rigidity and tearing thus being induced at a lower degree of penetration, as can also be concluded from Fig. 8. Contrarily, NR-S60 showed the latest signs of tearing and the smallest residual width in Fig. 8. This aligns with the material properties and lowest frictional forces stated above.

3.2 Correlation of friction to scratch behaviour

To draw further correlations between the material properties, scratch test data and the observed deformation transitions, the SEM results of Figs. 8 and 9 were combined with the data recorded during the scratch tests, along with images taken by both confocal and optical microscope. With these data, all transitions between the deformation modes (elastic deformation, ploughing, tearing, and cutting) could be accurately formed. For each material, the deformation modes are shown in Fig. 11, in which the frictional force generated during the scratch tests is plotted against the nominal contact area. The contact area was calculated using half the cone area corresponding to each penetration depth. A visual representation of the indenter-rubber contact is shown in

Fig. 12. Comparable representations were also reported by Xu et al. [5] and Briscoe and Sinha [34].

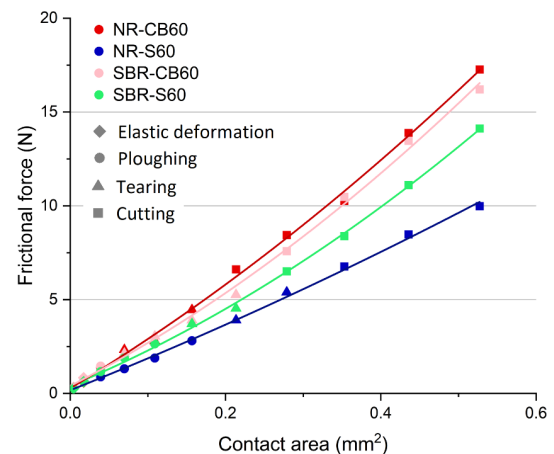


Fig. 11 The relation between the frictional force and the nominal contact area for all four materials in dry condition, including the deformation modes for each test.

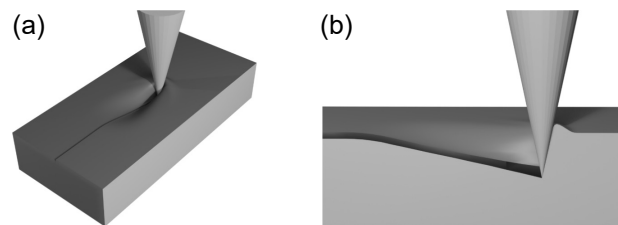


Fig. 12 A visual representation of the instantaneous contact between the indenter and the rubber during scratching.

Figure 11 shows a non-linear relation between the frictional force and contact area for rubber materials, due to the viscoelastic nature of rubber influencing the frictional force. However, for most non-rubber technical materials, the relation between the frictional force and contact area is linear. This relation between the frictional force and the contact area is related to the interfacial shear strength [26, 35]. It can also be concluded from Fig. 11 that for NR-CB60 cutting appears at the lowest contact area, hence the lowest penetration depth. The other three materials show cutting for larger contact areas; however, it shows that for all four materials, cutting occurs at similar frictional forces. It was also observed from Fig. 5(b) that the transition of tearing to cutting occurred at similar frictional forces in both dry and lubricated conditions. Both Figs. 5(b) and 10 highlight the impact of the deformation on the frictional force. These frictional forces and penetration depths connected to the transitions in deformation modes confirm the data visualised with the SEM, concluding that NR-S60 shows the lowest degree of damage after scratching. Comparing Fig. 11 with Figs. 8 and 9 shows the same relation as previously stated for Fig. 7: a higher frictional force at any contact area or penetration depth indicates worse scratch resistance for a material.

To continue the analysis, the deformation modes were correlated to the penetration depth in combination with the scratch performance. Figure 11 presents the transitions between deformation modes for the four materials. The slope of each curve represents the ratio between the frictional force and contact area, providing an indication of the shear strength. To further compare the deformation behaviour of the investigated compounds, Fig. 13 combines the transition depths with the corresponding frictional-force-to-contact-area ratios, thereby illustrating the deformation mode regimes, for each material.

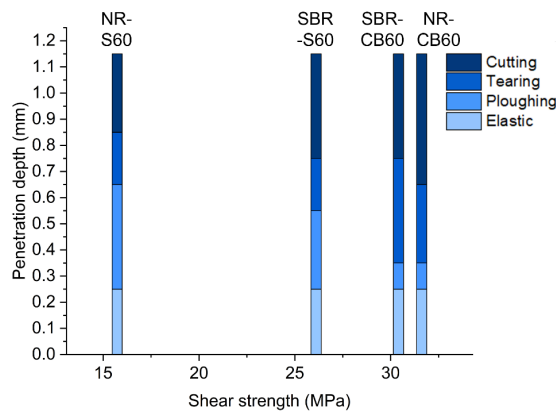


Fig. 13 Different deformation modes along the penetration depth versus the ratio between the frictional force and contact area for each material.

Figure 13 shows a few clear trends. Each deformation mode occurs at a lower penetration depth for NR-CB60 compared to the other materials. However, it can also be seen that the size of the ploughing mode region decreases with a decrease in scratch performance, as the transitions occur for lower penetration depths. It is expected that the elastic region also decreases for decreasing scratch resistance as the confocal data showed deformation closer to ploughing for NR-CB60 than for NR-S60 at a 0.2 mm penetration depth and a further stage of ploughing for NR-CB60 at 0.3 mm compared to NR-S60. Further investigations with step sizes much smaller than the 0.1 mm increments used in this study are needed to determine specific transition depths. For the materials exhibiting better resistance to scratch deformation (i.e., for materials which transition to cutting at higher penetration depths), the depth bandwidth in which the tearing mode is visible decreases, the ploughing mode regime becomes elongated towards higher penetration depths and the transitions in deformation modes occur for deeper penetration depths. Concluding Fig. 13, clear correlations were observed between the frictional force and the severity of the deformation behaviour showing that rubber compounds inducing higher frictional forces fail at lower penetration depths.

The results from Fig. 13 are specific to the test conditions employed, including the scratching speed, indenter geometry, and material. Additional experiments (not shown) demonstrated that

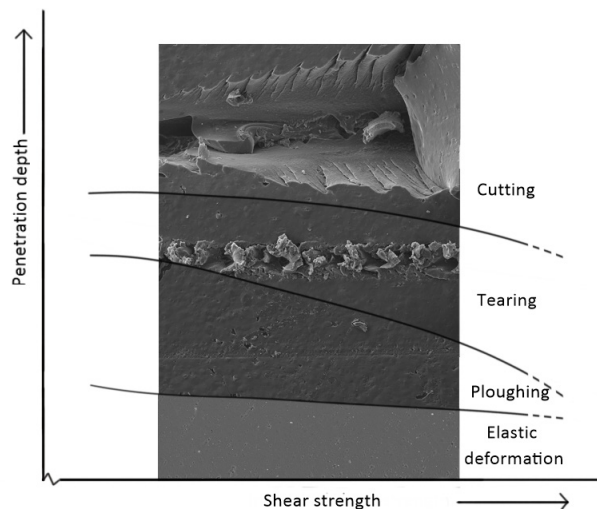


Fig. 14 The relation between the penetration depth and interfacial shear strength (ratio between the frictional force and contact area) independent of the rubber compound.

variations in these parameters shift the penetration depths and frictional-force-to-contact-area ratios at which deformation mode transitions occur. For example, decreasing the scratching speed or increasing the cone angle delayed the transitions, resulting in greater penetration depths before a given deformation mode was reached. Nevertheless, the same four deformation modes were consistently observed, indicating that they represent robust classifications of elastomer deformation behaviour and provide a framework for describing the early stages of wear evolution and the progressive damage processes that precede wear particle formation. To extend the applicability beyond the specific conditions investigated in this work, a generic deformation map is proposed in Fig. 14. It is hypothesised that other test conditions or rubber compounds can be incorporated within this framework, although further experimental validation is required.

4 Conclusions

This study showed deformation mode transitions of four rubber compounds, including NR or SBR and carbon black or silica, under single asperity scratch based testing conditions. The results confirmed the hypothesis that the deformation characteristics transition based on the contact conditions. The result is a proposed deformation mode map for rubber compounds, which were created to identify the characteristic regimes and transitions, which are shown based on different contact conditions. The test results show that four different wear deformation modes can be distinguished: elastic, ploughing, tearing, and cutting. The last mode represents that the total failure of the material was reached. The four different materials tested show disparate dependences on the penetration depth for all transitions and subsequent failure. These materials have different regimes of each mode, closely related to the frictional force. A rubber compound inducing higher shear strengths, for a certain penetration depth, was characterised by transitions, in deformation mode severity, occurring at lower depths.

The SEM images showed great similarities in cutting behaviour and stick-slip response within the compounds with the same base polymer. It could be concluded that a change in the amplitude of the signal correlates to the transition from tearing to cutting and that the compound showing the least damage, NR-S60, also showed the lowest frictional force for all penetration depths, indicating a strong relationship between the frictional force and the scratch performance. The SEM images also showed large differences between dry and lubricated conditions, in which all rubber compounds performed better in the lubricated conditions as could also be correlated to the frictional force, which was lower for lubricated conditions for similar penetration depths. Finally, it could be concluded that for dry and lubricated conditions and for the four different materials, failure was induced at similar frictional forces, which highlights the significant correlation between the frictional force and the damage of a rubber compound.

NR-S60 showed the least amount of damage, this compound is the material with the lowest strength and stiffness among the tested compounds, also showing the highest elongation at break. These factors combined most likely contribute to a better scratch resistance. Large similarities were identified in the severity of the damage of the compounds with the same filler type, where the carbon black compounds showed the worst scratch performance. Besides, the SEM images show a clear repetitive pattern for the NR-based compounds due to its strain induced crystallisation. Each ingredient therefore contributes differently to the deformational response of an elastomer to tribological conditions.

Finally, these results indicate that scratch-based testing can be a highly informative means to characterise deformation mode transitions of rubber compounds. Large differences were observed in deformation behaviour for small variations in compound composition, highlighting the effectiveness of this tool for screening new rubber grades.

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Competing interests

The authors have no competing interests to declare that are relevant to the content of this article.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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