

# Investigating the Wear Behaviors of Silane Coated Silica Filled Glass/Epoxy Nanocomposites

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## Abstract

Wear behaviors of silane coated silica (SiO<sub>2</sub>) nanoparticle-filled glass fiber reinforced polymer composites were investigated depending on the type of silane coating (KH550:  $\gamma$ -Aminopropyl-triethoxy-silane and KH570:  $\gamma$ -Methacryloxypropyl-trimethoxy-silane) and nanoparticle percentages within the polymer matrix. Unlike the cases given in most research studies, the silanized silica nanoparticles were used as received. Therefore, the synergistic effects of silanization and nanoparticle reinforcement were successfully applied since the silanization process was eliminated. The matrix modification with the nanoparticles was carried out using an ultrasound homogenizer, and then the modified matrix was reinforced with glass fibers. Scanned electron microscopy revealed the disperse ability of silanized silica nanoparticles within the glass/epoxy composites. The wear behaviors of the developed composites were investigated via a ball-on-disc tribology device. Wear track profiles were obtained depending on the width and depth of the wear. The length of wear track and wear mechanisms were determined by a stereomicroscope. The findings have shown that both types of silane coating and silica nanofiller percentages significantly affected the wear rate of the composite structures.

## Keywords

Glass fiber; Nano silica;  
Silane coating agents;  
Wear test; Wear  
mechanisms.

## Silan Kaplı Silika Dolgulu Cam/Epoksi Nanokompozitlerin Aşınma Davranışlarının Araştırılması

### Öz

Silan kaplı silika (SiO<sub>2</sub>) nanoparçacık dolgulu cam elyaf takviyeli polimer kompozitlerin aşınma davranışları, silan kaplama tipine (KH550:  $\gamma$ -Aminopropyl-triethoxy-silan, KH570:  $\gamma$ -Methacryloxypropyl-trimethoxy-silan) ve polimer matrisi içindeki nanoparçacık yüzdelere bağlı olarak incelenmiştir. Birçok araştırma çalışmasından farklı olarak, silanize silika nanoparçacıklar hazır bir şekilde kullanıldı. Bu nedenle silanizasyon işlemi ortadan kaldırdığı için silanizasyon ve nanoparçacık takviyesinin sinerjistik etkileri başarıyla uygulandı. Nanopartiküller ile matris modifikasyonu, ultrasonik homojenleştirici kullanılarak gerçekleştirildi ve ardından modifiye edilmiş matris, cam elyaflarla güçlendirildi. Taramalı elektron mikroskobu, cam/epoksi kompozitler içinde silanize silika nanoparçacıkların dağılılabirliğini göstermiştir. Geliştirilen kompozitlerin aşınma davranışları bir triboloji cihazı ile incelenmiştir. Aşınmanın genişliğine ve derinliğine bağlı olarak aşınma izi profilleri elde edilmiştir. Aşınma izinin uzunluğu ve aşınma mekanizmaları bir stereomikroskop ile belirlendi. Bulgular, her iki silan kaplama tipinin ve silika nanodolgu yüzdelere bağlı olarak aşınma durumunu önemli ölçüde etkilediğini göstermiştir.

### Anahtar Kelimeler

Cam elyaf; Nano silika;  
Silan kaplama ajanı;  
Aşınma testi; Aşınma  
mekanizmaları.

## 1. Introduction

Thermosetting fiber-reinforced polymer (FRP) composites, commonly including epoxy, have a low resistance to impact loads and show brittle failure behavior and poor tribological properties (Kwon *et*

*al.* 2017, Wu *et al.* 2015, Zhao *et al.* 2019). They also have poor thermal conductivity with high thermal expansion coefficients (Kausar 2020). By keeping the structural lightweight of the composites, the polymer matrix materials can be modified with

organic and inorganic particles (Deng *et al.* 2008) to enhance the structural performance of the FRP composites. Using secondary reinforcement elements like nano or microstructures can provide further improvements in the electrical, mechanical, physical, and thermal properties of the FRP composites (Bertasius *et al.* 2019, Liang and Wong 2017). Moreover, matrix strengthening can contribute to the out-of-plane properties of the composites (Shokrieh *et al.* 2015). The common additive materials that can be used as the secondary reinforcing constituents are carbon nanotube (Tang *et al.* 2013), graphene (Jia *et al.* 2018), silica (Panse *et al.* 2016), titania (Rubab *et al.* 2014), alumina (Mohanty and Srivastava 2015), and boron carbide (Geren *et al.* 2021).

The enhancements obtained by the matrix toughening with nano or microparticles are attributed to the reduced voids within the composite laminates, improved resistance to delamination failure of the laminates, and strong interfacial adhesion bonding between the fibers and the modified matrix (Kuzmin *et al.* 2016, Muralidhara *et al.* 2020, Wu *et al.* 2015). However, nanoparticles offer outstanding advantages even at very low contents than their microparticle counterparts (Elango and Faudzi 2015). Because nanoparticles have a high specific surface area that conducts a bridging effect, leading to better crack inhibition performance (Pun *et al.* 2019).

Silica (SiO<sub>2</sub>) nanoparticles are inorganic materials and have widespread usage due to their very low cost, high specific surface area, high-temperature resistance, and nontoxic features (Megahed *et al.* 2019, Pun *et al.* 2019, Wu *et al.* 2015). Lazar *et al.* (2018) used silica nanoparticles as a secondary reinforcement to improve the bending strength of glass FRP composites, and they obtained a nearly 40% increase at 0.75 wt.% of silica to epoxy resin ratio. Yıldırım *et al.* (Yildirim *et al.* 2017) increased the thermal decomposition temperature values of glass FRP composites with the addition of silica nanoparticles by about 45 °C. However, silica nanoparticles have poor disperse ability due to the susceptibility of their reaction with water

molecules, and thus the, agglomeration commonly becomes a challenging issue even at very low dispersion percentages (Saberian *et al.* 2019, Wang *et al.* 2019). The aggregation can also be attributed to the high surface energy of the nanoparticles (Ozsoy *et al.* 2017). The silica nanoparticles have been chemically treated with silane coupling agents to avoid agglomerations. The silane coatings can provide better dispersibility of the silica nanoparticles and conduct a bridging effect, obtaining higher mechanical and thermal properties (Allahverdi *et al.* 2012).

There are several commercial silane coupling agents such as KH550, KH560, KH570, and KH792 to functionalize the silica nanoparticles. A significant improvement in the adhesion can be achieved between the reinforcing fillers and the matrix material due to the presence of silane coupling agents (Panin *et al.* 2019). In the literature, many researchers have usually applied silanization process to eliminate the disadvantages of silica nanoparticles. Wu *et al.* (2015) used a KH550 type silane coupling agent and highly reduced the agglomeration of the nanoparticles within the epoxy matrix. Zhang *et al.* (2021) compared the effects of different silane coupling agents on the mechanical and thermal properties of the polymer matrix. The results showed that the KH550 and KH792 types of silane coatings on silica nanoparticles provided significant improvements compared to the KH560 and KH570 types. Wang *et al.* (2019) also reported the better integration of KH550 considering the thermodynamical properties of silanized SiO<sub>2</sub>/cellulose composites.

The polymer matrix composites reinforced with several fibers and additive materials as fillers have been subjected to wear in many engineering applications. Therefore, investigations of the reinforced composites with modified matrix materials on the tribological properties have also attracted researchers. Zhao *et al.* (2019) reported a considerable reduction in coefficient of friction (COF) and wear rate values of epoxy composites by incorporating nano-sized boron nitride filler. Ozsoy *et al.* (2017) dispersed microparticles (alumina,

titania, and fly ash) into the epoxy and improved the COF and specific wear rates; 20 wt.% of fly ash micro fillers provided the best reductions. Silane coupling agents also play an important role to achieve better anti-wear properties. Panin *et al.* (2019) compared two silane-containing modifiers (Penta 1006 and KH550) for the wear properties of chopped glass fiber-filled ultrahigh molecular weight polyethylene composites. They reported that KH550 provided more effective compatibility with the glass fibers, so better tribological properties were obtained.

In this study, the effects of as received silane coated silica nanoparticles on the wear behaviors of nano-silica filled glass FRP composites were investigated. Both KH550 ( $\gamma$ -Aminopropyl-triethoxy silane) and KH570 ( $\gamma$ -Methacryloxypropyl-trimethoxy silane) silane coated silica nanoparticles were dispersed within epoxy at a weight ratio of 1.5% and 3%, respectively. Then the modified polymer matrix was reinforced with plain-woven glass fibers, acting as the primary reinforcement. The wear loss, specific wear rate, and wear mechanisms of the developed composites were evaluated considering the type of silane coating and nanofiller percentages.

## 2. Materials and Method

### 2.1 Composite Constituents and Manufacturing

The developed composites have consisted of plain-woven glass fibers, epoxy resin set, and silanized silica ( $\text{SiO}_2$ ) nanoparticles. Glass fibers have 2560  $\text{kg/m}^3$  density, 3400 MPa tensile strength, 73 GPa tensile modulus, and the elongation at break of the fibers is 2.75 %. Epoxy resin set consists of L160 epoxy resin and H160 hardener, which the materials were mixed at a weight ratio of 100:25. The polymer matrix has a density of about 1190  $\text{kg/m}^3$ , and the tensile, compressive, and impact strength values are

approximately 75 MPa, 90 MPa, and 45  $\text{kJ/m}^2$ , respectively. The elongation at break of the matrix is between 5-6.5%. The silanized  $\text{SiO}_2$  nanoparticles have a particle size of 18-35 nm and a specific surface area of 150-550  $\text{m}^2/\text{gr}$ . The types of silane coatings were KH550 ( $\gamma$ -Aminopropyl-triethoxy silane) and KH570 ( $\gamma$ -Methacryloxypropyl-trimethoxy silane). The amount of silane coatings for both KH550 and KH570 is 3-4% with respect to the amount of silica. The purity of silane-coated  $\text{SiO}_2$  nanoparticles is above 95.9% and 96.3% for KH550 and KH570, respectively. All these specifications were obtained from the suppliers. The nanoparticles were used as received; therefore, the silane treatment process of silica nanoparticles was eliminated.

The nanocomposites were manufactured in two stages. Once the modified polymer matrix materials were obtained and then they were reinforced with glass fibers. The steps of the manufacturing process are presented in Figure 1.

### 2.2 Microscopic Examinations

Scanned electron microscopy (SEM) and stereomicroscope were utilized in the present study. The SEM was used to analyze the dispersibility of silanized  $\text{SiO}_2$  nanoparticles within the glass/epoxy composites. The observations were made via Quanta 650 Field Emission SEM device, and to improve the image quality a very thin conductive coating material was applied over the specimens. The stereomicroscope was used via Nikon SMZ1500 stereomicroscope device to measure the wear track's length and determine the wear mechanisms on the worn surfaces.

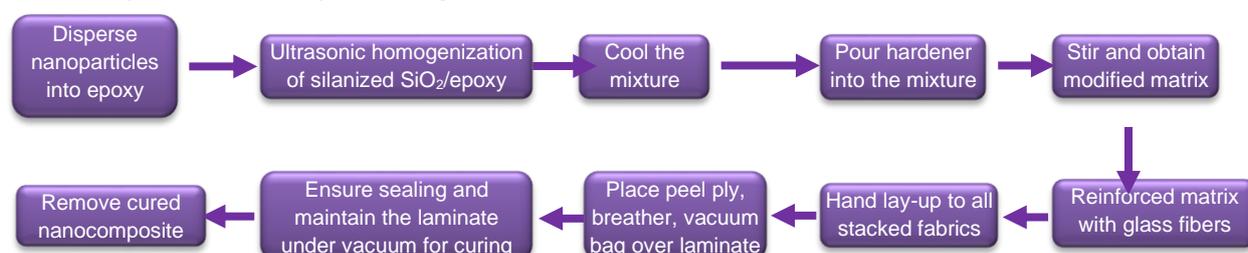


Figure 1. The manufacturing process of the silanized silica filled glass FRP nanocomposites

### 2.3 Wear Test

The wear tests were carried out to reveal the wear behavior of the silanized nano silica filled glass FRP composites. The investigations were made by utilizing a ball-on-disc type multi-functional tribometer testing device (UTS Tribolog /www.uts-design.com, Turkey). The tests were performed as dry rotating for a sliding distance of 400 m under 35 N normal load for approximately one hour. The rotating speed was 125.6 mm/s and the ball material was 6 mm diameter Al<sub>2</sub>O<sub>3</sub>. During the wear tests, wear track profiles of the hybrid composites were obtained according to the scanned distance and wear depth.

To compare the rate of wear, the volume loss (V) of the nanocomposites was determined. V values were calculated based on the product of integrated area of the wear track profiles and the average track length (L). This procedure was also reported in the literature (Aguilera-Camacho *et al.* 2021). Then the specific wear rate can be calculated according to Equations (1) and (2) (Aguilera-Camacho *et al.* 2021).

$$V = A_{wt} \times L \quad (1)$$

$$K = \frac{V}{F.S} \quad (2)$$

Where V is the volume loss (mm<sup>3</sup>), A<sub>wt</sub> is the wear track integrated area (mm<sup>2</sup>), L is the track length (mm) that is measured on the worn surface with the aid of a stereomicroscope, K is the specific wear rate (mm<sup>3</sup>/Nm), F is the applied load (N), and S is the sliding distance (m).

## 3. Results and Discussion

### 3.1 Analysis of Silanized Silica Nanoparticles' Dispersibility

The dispersibility of silanized silica nanoparticles within the polymer matrix were examined with SEM images. Figure 2 presents the glass FRP (GFRP) composites hybridized with silanized silica nanoparticles depending on the type of silane coating and filler percentages. The sizes of some detected nanoparticles were measured, and the values are given in Figure 2.

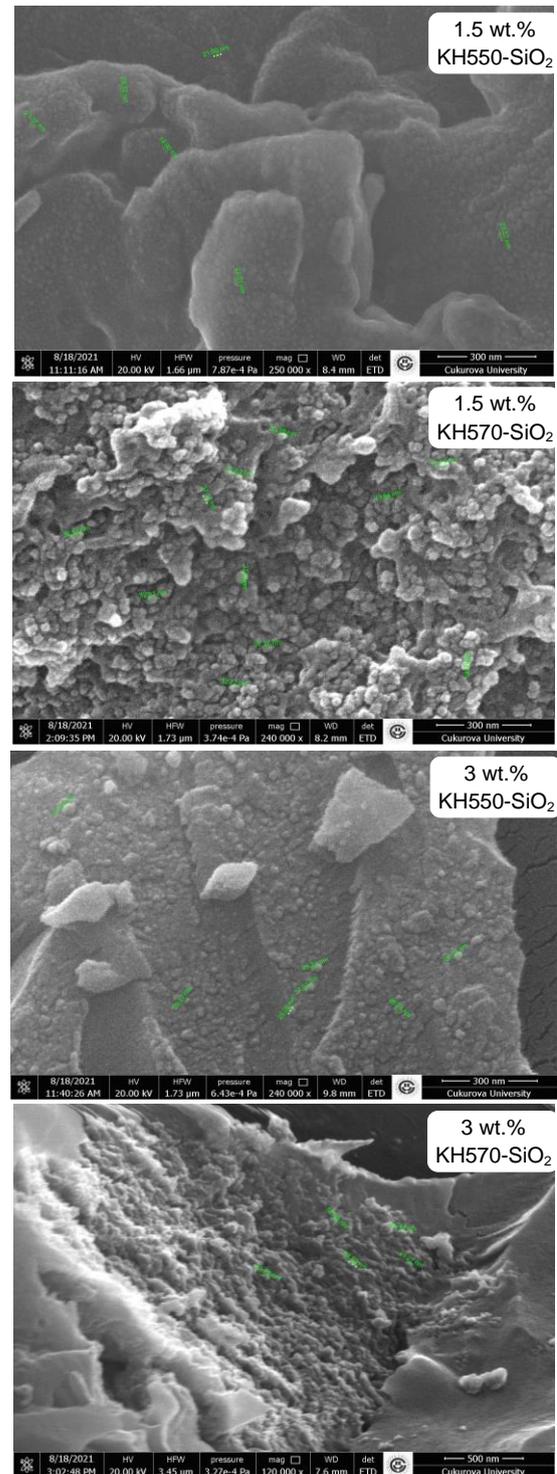
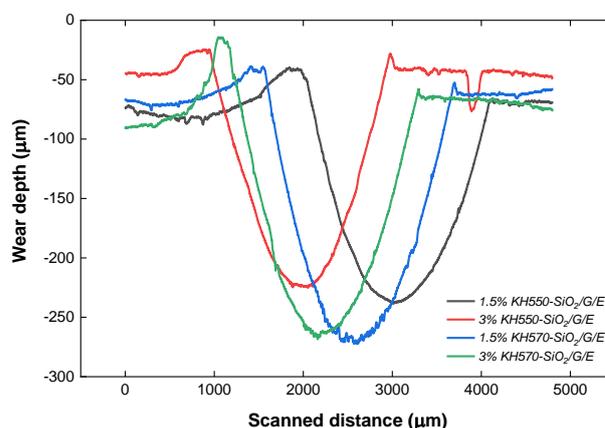


Figure 2. SEM images of the silanized silica filled GFRP nanocomposites

According to the supplier's datasheet, the size of nanoparticles is in the range of 18–35 nm. As shown in Figure 2a (1.5 wt.% KH550 SiO<sub>2</sub> filler addition) and 2c (3 wt.% KH550 SiO<sub>2</sub> filler addition), nanoparticles' sizes are below 35 nm. However, the sizes of some nanoparticles are around 35 nm in Figure 2b (1.5 wt.% KH570 SiO<sub>2</sub> filler addition), and the sizes of some nanoparticles are above 35 nm in Figure 2d (3 wt.% KH570 SiO<sub>2</sub> filler addition). It is evident that the KH550 silane type has provided better integrity with the polymer matrix, so improved adhesion bonding was achieved. In the literature, Wang *et al.* (2020) functionalized the glass/PVC composites with KH550, KH560, and KH570. Their reported SEM images showed that the KH550 type of coating provided the best interfacial bonding of the glass fibers. On the other hand, the use of KH570 type silane coating did not finely homogenize the nanoparticles into smaller grains at 1.5 wt.% filler addition and did not prevent agglomeration at 3 wt.% filler addition. The poor dispersibility adversely affects the load transfer between the matrix and the reinforcements leading to weakness in mechanical properties (Ulus *et al.* 2016, Zheng and Park 2019).

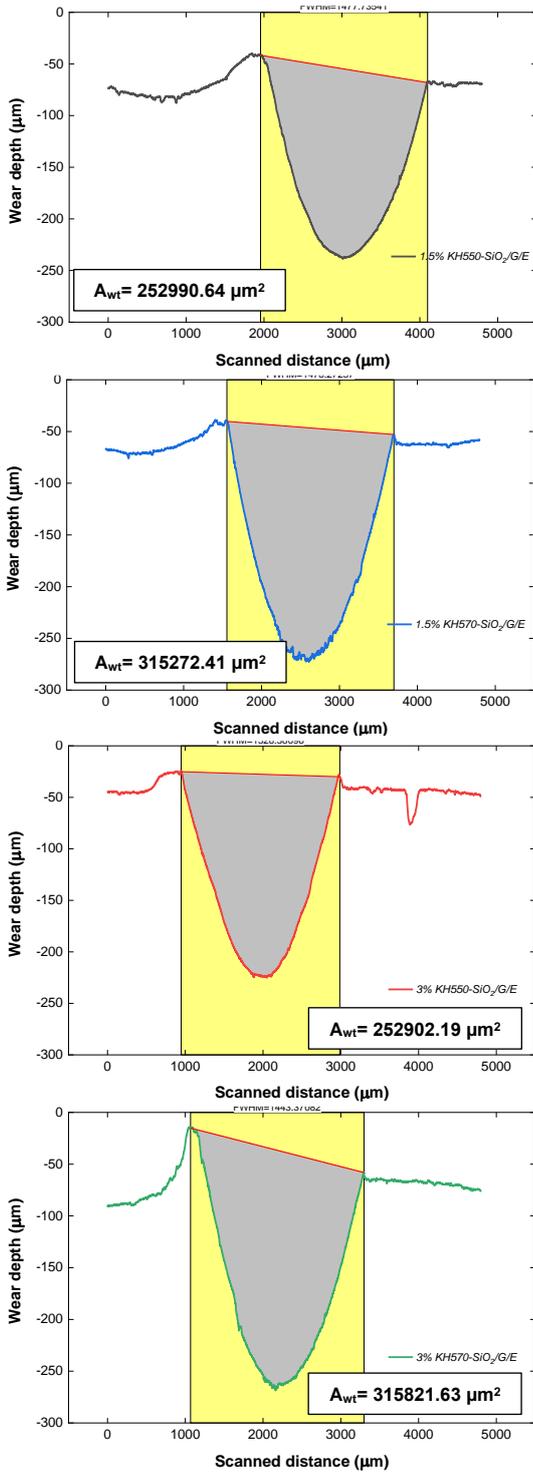
### 3.2 Wear Behavior

The wear resistances of the nanocomposites were revealed based on the wear track profiles. The wear depth versus scanned distance curves, given in Figure 3, comparatively presents the wear track profiles of the nanocomposites. Both the type of silane coating and nanoparticle percentages have significantly affected the wear behaviors. KH550 coated silica filled GFRP nanocomposite has provided lower wear depth than that of KH570 coating, irrespective of filler percentages. On the other hand, the maximum depth of wear values of 1.5 wt.% silica filled GFRP nanocomposites were obtained at higher scanned distance values than that of 3 wt.% silica filled GFRP nanocomposites, irrespective of silane coating type. It can be inferred that increasing the filler percentages has caused the hybrid composite to be worn earlier.



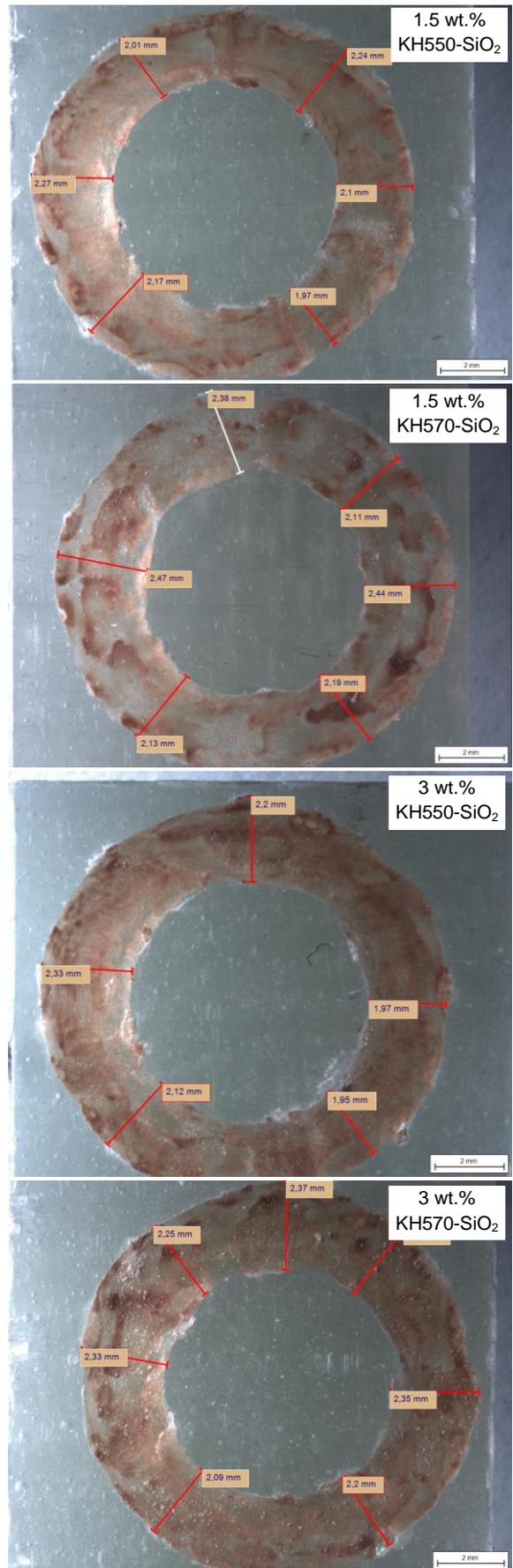
**Figure 3.** Wear track profiles of silanized SiO<sub>2</sub> filled GFRP nanocomposites.

The area ( $A_{wt}$ ) under the curves was shaded, as shown in Figure 4. It can be seen that the area obtained from both 1.5 wt.% and 3 wt.% filler addition was found to be nearly the same for each silane coating. However, KH550-SiO<sub>2</sub> fillers have provided the GFRP nanocomposite to obtain a lower track area than that of KH570-SiO<sub>2</sub> fillers, irrespective of filler percentages. Because the depth of wear was observed higher in the case of KH570 silane coating. This could be due to the better integration of the KH550 silane coating agent for SiO<sub>2</sub> nanoparticles leading to improved interlaminar adhesion between the glass fibers and the modified matrix. Therefore, the resistance of KH550 coated silica-filled GFRP nanocomposites against wear was better, leading to a lower depth of wear. Panin *et al.* (2019) also recommended applying silane coating of KH550 for better anti-wear properties. On the other hand, the higher depth of wear obtained with KH570 coated silica-filled GFRP nanocomposites could be attributed to the non-uniform distribution of the nanoparticles, leading to poor interfacial strength between the glass fiber and the matrix. The agglomerations can weaken the load transfer and accelerates the composites' failure due to the stress concentration around the agglomerates. Guo *et al.* (2019) compared the silanization effects of KH550 and KH570 and reported that KH550 had more impact on the conducting of strong adhesion bonding that led to improved wear resistance.



**Figure 4.** The area under the wear track profile ( $A_{wt}$ ) of the silanized silica filled GFRP nanocomposites.

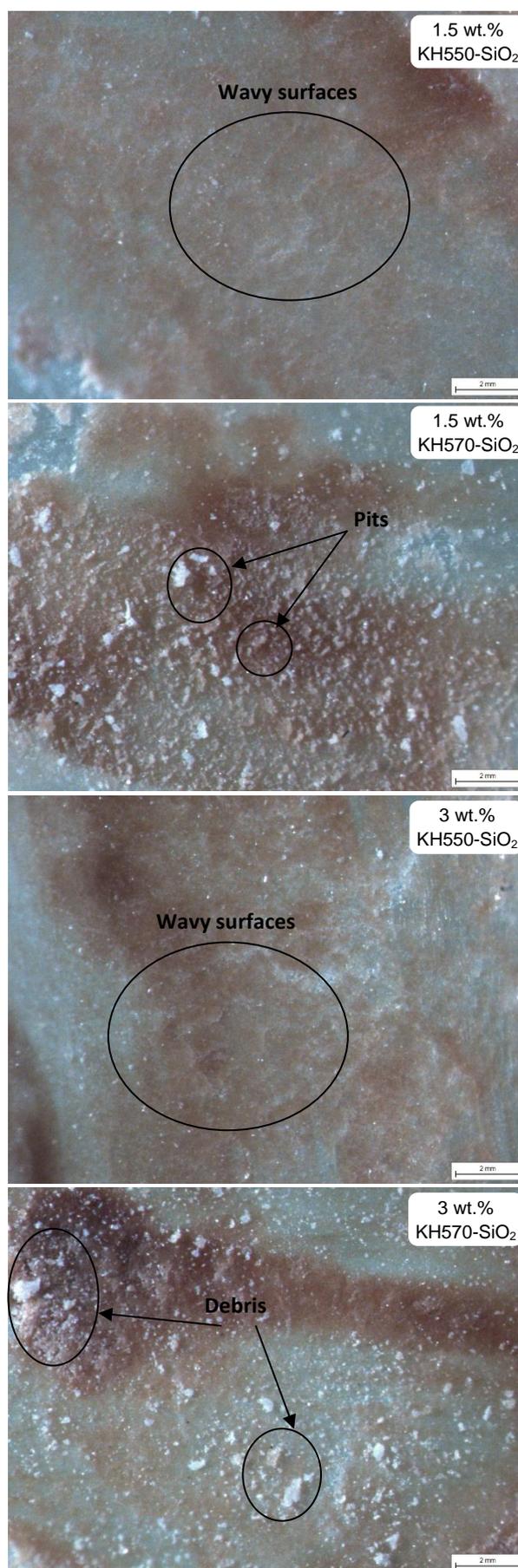
The length of wear track values of the nanocomposites was obtained using a stereomicroscope. As seen in Figure 5, the track length was measured on the worn surfaces and recorded from several locations. The average L values and their standard deviations are written in Table 1.



**Figure 5.** Measurements of wear track length of the silanized silica filled GFRP nanocomposites.

When Table 1 is scrutinized, the approximate wear track area ( $A_{wt}$ ) was found 0.25 mm<sup>2</sup> and 0.32 mm<sup>2</sup> for KH550 and KH570 coated silica GFRP nanocomposites, respectively, and there is no significant difference between the nanocomposites having various filler percentages for each of the silane coating types. Similar trend can also be seen for other parameters in Table 1. The wear rate values were approximately found  $38 \times 10^{-6}$  mm<sup>3</sup>/Nm and  $52 \times 10^{-6}$  mm<sup>3</sup>/Nm for KH550 and KH570 coated silica GFRP nanocomposites, respectively. KH570 silane coating for silica nanoparticles has caused about 36-38% higher volume loss and specific wear rate compared to the results of KH550.

The wear failure mechanisms of the nanocomposites were examined on the worn surfaces using the stereomicroscope. Plastic deformation and adhesion are the typical wear mechanisms observed mainly for the KH550 silane coated silica GFRP nanocomposites, as seen in Figures 6a and 6c. Because the worn surfaces are seen wavy. The failure mechanisms for KH570 silane coated silica GFRP nanocomposites (Figures 6b and 6d) were developed in a severe condition compared to the KH550. Also, wear debris particles, and pit formations were more visible in Figures 6b and 6d. Similar wear mechanisms were observed by Sharma *et al.* (2020).



**Figure 6.** Stereomicroscope examination of wear mechanisms of silanized silica filled GFRP nanocomposites.

**Table 1.** Wear results of the silanized silica filled GFRP nanocomposites.

| Nanocomposites                       | A <sub>w</sub> t (mm <sup>2</sup> ) | L (mm)        | V (mm <sup>3</sup> ) | K (mm <sup>3</sup> /Nm) x 10 <sup>-6</sup> |
|--------------------------------------|-------------------------------------|---------------|----------------------|--|
| 1.5 wt.% KH550-SiO <sub>2</sub> /G/E | 0.25299                             | 2.098 (0.099) | 0.531                | 37.91                                      |
| 3 wt.% KH550-SiO <sub>2</sub> /G/E   | 0.25290                             | 2.114 (0.142) | 0.534                | 38.19                                      |
| 1.5 wt.% KH570-SiO <sub>2</sub> /G/E | 0.31527                             | 2.322 (0.136) | 0.732                | 52.29                                      |
| 3 wt.% KH570-SiO <sub>2</sub> /G/E   | 0.31582                             | 2.300 (0.064) | 0.726                | 51.89                                      |

#### 4. Conclusion

The effects of nano-silica percentages and type of silane coating over the nanoparticles on the glass FRP composites have been revealed based on the wear behaviors. Both the filler amount and silane type have significant effects on the results. Mainly, the following conclusions can be drawn from the study:

- Silane coating of silica nanoparticles with KH550 has provided better dispersibility than KH570.
- The wear grooves have become broader and deeper for KH570 coated silica-filled GFRP nanocomposites. The length and area of wear track values were found to be approximately 10% and 25% higher when compared to that of KH550 coated silica-filled GFRP nanocomposites.
- *Increasing the silanized silica filler percentages (irrespective of silane type) led to the wearing out of GFRP nanocomposites at relatively lower scanned distance values.*
- The volume loss and specific wear rate values of KH550 coated silica-filled GFRP nanocomposites were 36-38% lower than that of KH570, irrespective of the filler percentages.
- While the wear debris particles and pit formations were the dominant wear mechanisms for KH570 coated silica-filled GFRP nanocomposites, the waviness of the worn surfaces observed for KH550 coated silica filled GFRP nanocomposites has indicated the wear mechanisms of plastic deformation and adhesion.

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