SURFACE STICKINESS AND WAVINESS OF TWO-LAYER SILICONE STRUCTURES FOR SYNTHETIC VOCAL FOLDS

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Abstract: Body-cover models of vocal folds made of silicone rubber are increasingly used in voice research. The very soft cover layer often shows a residual stickiness. This study explored potential methods to avoid the stickiness while maintaining the softness and a flat surface structure. Therefore, 60 silicone samples were fabricated from different silicones and with different manufacturing procedures to simulate the compound of cover layer and epithelium layer. 30 silicone samples were tempered in different ways and 30 samples were powdered between the two layers. The fabricated samples were investigated with respect to their stickiness and surface waviness. It could be shown that some materials and procedures are better suited than others to produce synthetic vocal folds that are as little sticky as possible and have a flat surface at the same time.

1 Introduction

Self-oscillating silicone models are increasingly used to study the behaviour of human vocal folds, because silicone rubber can imitate the tissue properties, and especially the Young's modulus, of real vocal folds well. In order to create realistic models of vocal folds, it is important to reproduce their layered structure. The vocalis muscle is represented by a body layer, and the lamina propria by a cover layer. The lamina propria is extremely soft and can also be covered by a very thin, stiffer epithelial layer, as is the case with real vocal folds. The effects of the epithelial layer on phonation is not yet fully understood, but it has been shown that an epithelial layer leads to a better glottal closure [1, 2].

The softness of the cover layer is achieved by mixing high amounts of thinner, i.e., silicone oil, into the silicone composite, which leads to stickiness of the vulcanized silicone. Covering with an epithelial layer, to which no thinner is added, does not significantly reduce the stickiness of the models. According to the manufacturer, the oil diffuses out of the cover layer over a long period of time and into the epithelial layer above. In order to reduce the stickiness, talcum powder is usually applied on top of the epithelial layer in preparation for experiments [3, 4].

Preliminary tests with silicones from different manufacturers and different shore hardnesses have shown that the epithelial layer is the less sticky the thicker the layer or the harder the silicone is. However, the application of harder or more viscous non-vulcanized silicone mixtures results in waviness after vulcanization of the epithelial layer, which alters the surface shape. There was also evidence of reduced stickiness when the individual layer were tempered. According to the silicone manufacturer, a tempering process accelerates the diffusion of the oil inside. Further preliminary tests indicated that the introduction of powders between the cover layer and the epithelial layer can reduce the stickiness, too.

For these reasons, we have carried out a systematic study where we analysed the surface waviness and stickiness of 60 silicone samples made of different silicones and with different manufacturing processes. Jars as sample containers were filled with a silicone for the basis matter, corresponding to the cover layer, on top of which different silicones were added as epithelial layer. The stiffness of the basis matter was modelled to correspond to the transversal stiffness of the lamina propria of human vocal folds with a Young's modulus (E) of around 1.2 kPa [5]. The surface waviness was evaluated by visual inspection and the stickiness was determined by an adhesion experiment with aluminium cylinders of different weights.

2 Methods

The 60 silicone samples produced differed in terms of the silicones used for the basis matter and the epithelial layer, in terms of tempering of the layers, and the insertion of a powder layer. The layers of 30 silicone samples were partially tempered to determine the influence of tempering. These are subsequently referred to as "tempered silicone samples". In addition, 30 silicone samples were produced in which powder was inserted between the basis matter and the epithelial layer to investigate a possible reduction in stickiness. These are called "silicone samples with powder layer" in the following.

2.1 Materials

Based on our preliminary investigations the following materials were used:

- TP: Talkum Powder for general purpose, ($Mg_3H_2(SiO_3)_4$), CAS 14807-96-6
- Ab: Acetylen Black Carbon Powder, compressed, 42nm particle diameter, CAS 1333-86-4
- AP: ABSODAN Plus Diatomaceous Earth, oil binding agent Type III/R, 0.5 - 1mm particle diameter, grinded
- EF6: SmoothOn Ecoflex 0030 (1 part A : 1 part B) plus SmoothOn silicone thinner (6 parts)
- TF4: TrollFactory TFC Silikon Kautschuk Typ 13 Shore 00 (1 part A : 1 part B) plus SmoothOn silicone thinner (4 parts)
- TFG: TrollFactory TFC Silikon Kautschuk Typ 20 supersoft
- TF00: TrollFactory TFC Silikon Kautschuk Typ 13 Shore 00 (1 part A : 1 part B)
- TF20: TrollFactory TFC Silikon Kautschuk Typ 1 Shore 20 (1 part A : 1 part B)
- EF00: SmoothOn Ecoflex 0030 (1 part A : 1 part B)

2.2 Tempered silicone samples

The silicone samples consisted of two layers: the basis matter layer representing the vocal fold cover and the epithelial layer. The sample containers were first filled with the silicones for the basis matter and cured for over 24 h at room temperature. As next step, one half of the sample containers were tempered for over 3 h at 120°. Then the epithelial layer was applied by putting a small amount of the non-vulcanized silicone mixture on top of the basis matter. The surface of the sample container was then immediately aligned vertically allowing the silicone to drain off over 5 min such that only a very thin layer remained. After that one half of the samples were tempered again for over 3 h at 120°. In order to further reduce the stickiness, a second layer of the epithelial layer was applied using the same procedure to most of the samples, some of which were tempered again. An overview of the prepared samples and the tempering steps is shown in Table 1.

Table 1 – Overview of the tempered silicone samples.

Sample	Material basis matter	Young's modulus	Basis matter tempered	Material cover	Young's modulus	First layer tempered	Second layer tempered
01	EF6	1.1 kPa	No	TF00	56 kPa	No	No
02	EF6	1.1 kPa	No	TF00	56 kPa	Yes	Yes
03	EF6	1.1 kPa	No	TF20	560 kPa	No	No
04	EF6	1.1 kPa	No	TF20	560 kPa	Yes	Yes
05	EF6	1.1 kPa	Yes	TF00	56 kPa	No	No
06	EF6	1.1 kPa	Yes	TF00	56 kPa	Yes	Yes
07	EF6	1.1 kPa	Yes	TF20	560 kPa	No	No
08	EF6	1.1 kPa	Yes	TF20	560 kPa	Yes	Yes
25	EF6	1.1 kPa	Yes	TF00	56 kPa	Yes	—
26	EF6	1.1 kPa	Yes	TF00	56 kPa	Yes	—
09	TF4	1.2 kPa	No	TF20	560 kPa	No	No
10	TF4	1.2 kPa	No	TF20	560 kPa	Yes	Yes
11	TF4	1.2 kPa	No	TF00	56 kPa	No	No
12	TF4	1.2 kPa	No	TF00	56 kPa	Yes	Yes
13	TF4	1.2 kPa	Yes	TF20	56 kPa	No	No
14	TF4	1.2 kPa	Yes	TF20	560 kPa	Yes	Yes
15	TF4	1.2 kPa	Yes	TF00	56 kPa	No	No
16	TF4	1.2 kPa	Yes	TF00	56 kPa	Yes	Yes
27	TF4	1.2 kPa	Yes	TF20	560 kPa	Yes	—
28	TF4	1.2 kPa	Yes	TF20	560 kPa	Yes	—
17	TFG	4.5 kPa	No	TF00	56 kPa	No	No
18	TFG	4.5 kPa	No	TF00	56 kPa	Yes	Yes
19	TFG	4.5 kPa	No	TF20	560 kPa	No	No
20	TFG	4.5 kPa	No	TF20	560 kPa	Yes	Yes
21	TFG	4.5 kPa	Yes	TF00	56 kPa	No	No
22	TFG	4.5 kPa	Yes	TF00	56 kPa	Yes	Yes
23	TFG	4.5 kPa	Yes	TF20	560 kPa	No	No
24	TFG	4.5 kPa	Yes	TF20	560 kPa	Yes	Yes
29	TFG	4.5 kPa	Yes	TF00	56 kPa	Yes	—
30	TFG	4.5 kPa	Yes	TF20	560 kPa	Yes	—

2.3 Silicone samples with powder layer

In order to find out whether the insertion of an intermediate layer of oil-absorbing powder can reduce the stickiness and waviness, 30 additional silicone samples with an intermediate layer of different powders were prepared. The jars used as sample containers here are very similar to those in Section 2 but have a slightly larger volume (26 ml compared to 18 ml) and a 2 mm smaller opening diameter (28.5 mm vs. 30.5 mm). The volume difference was compensated by partially filling the jars with TF20 enclosing gravel sand. The very small difference in the opening area was considered as insignificant. Furthermore, tempering was completely omitted, because it represents a high additional effort.

As with the tempered silicone samples, the first step was to produce the basis matter by filling the sample containers with silicone. The mixture vulcanized for 48 h at room temperature. One out of five samples remained as reference, while the others were powder-coated. One group of samples was coated with a thin layer of talcum powder by scattering a small amount onto the basis matter and rubbing it with a glove (Vinyl, powder-free) in circular motions till the silicone stopped sticking to the glove. To another group of samples a thick powder layer was applied by dipping the complete jar surface into talcum powder. The surplus powder on the surface was removed by tapping the jar. Other groups of samples were coated with acetylene black carbon powder and ABSODAN Plus Diatomaceous Earth powder equivalent to the procedure with thin talcum powder. The particles of AP needed to be grinded before the application. After applying the powder coating, an epithelial layer was applied to the silicone samples according to the procedure of the tempered samples and cured at room temperature over 24 h. An overview of the silicone samples with a powder layer can be found in the Table 2.

Table 2 – Overview of the silicone samples with different powder layers between the basis matter and the surface layer.

Sample	Material basis matter	Young's modulus	Powder Layer	Thickness	Material cover	Young's modulus
31	EF6	1.1 kPa	None	-	EF00	67 kPa
32	EF6	1.1 kPa	Talkum	Thin	EF00	67 kPa
33	EF6	1.1 kPa	Talkum	Thick	EF00	67 kPa
34	EF6	1.1 kPa	Acetylen Black	Thin	EF00	67 kPa
35	EF6	1.1 kPa	Absodan Plus	Thin	EF00	67 kPa
36	EF6	1.1 kPa	None	-	TF00	56 kPa
37	EF6	1.1 kPa	Talkum	Thin	TF00	56 kPa
38	EF6	1.1 kPa	Talkum	Thick	TF00	56 kPa
39	EF6	1.1 kPa	Acetylen Black	Thin	TF00	56 kPa
40	EF6	1.1 kPa	Absodan Plus	Thin	TF00	56 kPa
41	EF6	1.1 kPa	None	-	TF20	560 kPa
42	EF6	1.1 kPa	Talkum	Thin	TF20	560 kPa
43	EF6	1.1 kPa	Talkum	Thick	TF20	560 kPa
44	EF6	1.1 kPa	Acetylen Black	Thin	TF20	560 kPa
45	EF6	1.1 kPa	Absodan Plus	Thin	TF20	560 kPa
46	TF4	1.2 kPa	None	-	EF00	67 kPa
47	TF4	1.2 kPa	Talkum	Thin	EF00	67 kPa
48	TF4	1.2 kPa	Talkum	Thick	EF00	67 kPa
49	TF4	1.2 kPa	Acetylen Black	Thin	EF00	67 kPa
50	TF4	1.2 kPa	Absodan Plus	Thin	EF00	67 kPa
51	TF4	1.2 kPa	None	-	TF00	56 kPa
52	TF4	1.2 kPa	Talkum	Thin	TF00	56 kPa
53	TF4	1.2 kPa	Talkum	Thick	TF00	56 kPa
54	TF4	1.2 kPa	Acetylen Black	Thin	TF00	56 kPa
55	TF4	1.2 kPa	Absodan Plus	Thin	TF00	56 kPa
56	TF4	1.2 kPa	None	-	TF20	560 kPa
57	TF4	1.2 kPa	Talkum	Thin	TF20	560 kPa
58	TF4	1.2 kPa	Talkum	Thick	TF20	560 kPa
59	TF4	1.2 kPa	Acetylen Black	Thin	TF20	560 kPa
60	TF4	1.2 kPa	Absodan Plus	Thin	TF20	560 kPa

2.4 Measurements

The properties of the tempered silicone samples were determined about 3 weeks after their preparation, and the properties of the samples with powder layer were determined nine days after preparation.

2.4.1 Young's modulus

To determine the Young's moduli (E) of the silicon materials, rod-like specimens with a diameter of 4.7 mm and length of 50 mm were manufactured. E was determined from the linear slope of the stress-strain curves obtained within a quasi-static elongation of the rods utilizing a MCR 301 rheometer by Anton Paar. The rate of deformation in the measurements was set as 0.5 mm/s. Hereby, maximum elongation of the tested specimens was limited to 5 % in order to avoid a significant change of the specimens geometry. The determined moduli are given in Tables 1 and 2.

2.4.2 Waviness

To determine the waviness, a visual inspection was carried out and a score of 0 to 2 was given. The score 2 corresponds to waviness over the entire surface, score 1 corresponds to partial waviness of the surface, and score 0 represents a smooth surface. Examples for the given score are shown in Figure 1. The white, gray and black circles indicate no waviness (0), partial waviness (1) and total waviness (2), respectively.

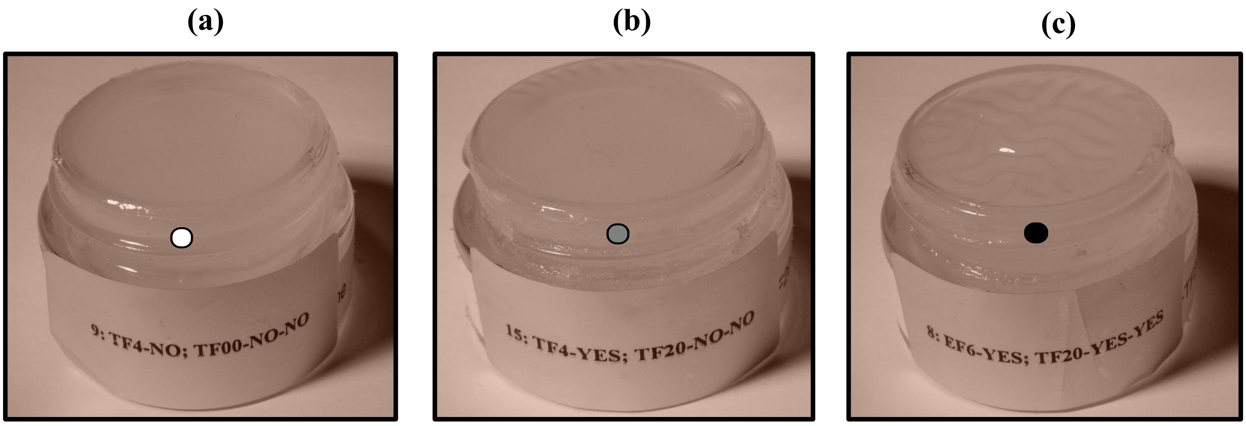

Figure 1 – Examples of waviness of the silicone samples – (a) Smooth surface; (b) Slightly deformed surface; (c) Strongly deformed surface

2.4.3 Stickiness

The stickiness was determined by counting how often aluminium cylinders of different weights sticked to the surface of the samples for a certain duration over five passes. The aluminium cylinders had a diameter of 12 mm and were manufactured in different heights/weights. A total of 8 cylinders with weights from 1 g to 2.4 g and a weight increment of 0.2 g were used. Each silicone samples was clamped in a holder with the surface layer facing down. Then, starting with the heaviest weight, the weight was pressed with some pressure against the surface of the silicone. If the weight did not drop for more than 10 s this was counted as a stick event. If the weight dropped before 10 s expired, this was not counted as stick event. The experiment was repeated five times and for each weight the stick events were added. A picture of the measurement setup can be seen in Figure 2.

Figure 2 – Picture of stickiness measurement setup.

3 Results and Discussion

3.1 Waviness

As displayed in Figures 3 and 4, all models with the TF20 surface layer exhibit increased waviness. This might has two reasons. First, the silicones TF20 and EF00 are more viscous than TF00, in non-vulcanized state, which leads to *thicker* layers of the epithelium. Secondly, TF20 has a much higher E which can lead to higher stresses within the silicone and thus cause the waviness. However, since the non-vulcanized silicone mixture EF00 subjectively has a similar viscosity as TF20 but does not tend to form waves, the most probable reason for strong waviness is the difference in stiffness between the basis matter an the epithelial layer.

3.2 Stickiness

As shown in Figure 5, the tempered silicone samples made of basis matter EF6 have the lowest stickiness compared to all other tempered silicone samples, probably due to a better oil absorption ability. Sample 02 shows the best result because it has a smooth surface with the least stickiness. The low stickiness of models with increased waviness is not necessarily due to the material properties. The low adhesion of the aluminum cylinders is due to the stresses in the material, caused by the waviness, after attaching the cylinders. This is also evident within the silicone samples with powder layer, see Figure 6. The stickiness of the samples with the basis matter made of TF4 and TFG is at the upper end of the measuring range, so no statement can be made about the influence of tempering. Tempering of the samples with EF6 basis matters does not lead to any improvement in stickiness. A slight improvement can only be observed in the tempering of the TF00 surface layers when samples 01 and 02 are compared.

The powder layer reduced the stickiness of all silicone samples in the cases where a smooth surface is present, see Figure 6. All reference bodies without powder have the maximum stickiness which can be quantified in the experimental setup. [Ab] and [AP] also improve stickiness. However, due to the clumping and damage of the base body surface during rubbing, they have in some cases considerable inhomogeneities in the surface stickiness, which can not be fully averaged out by the measuring method. The sample 40 shows a strong reduction of the stickiness, which cannot be explained in comparison to the lower improvement within sample 50 at the moment. The lowest stickiness with a smooth surface was found in the samples 48 and 49 with a basis matter of TF4 and a surface layer of EF00.

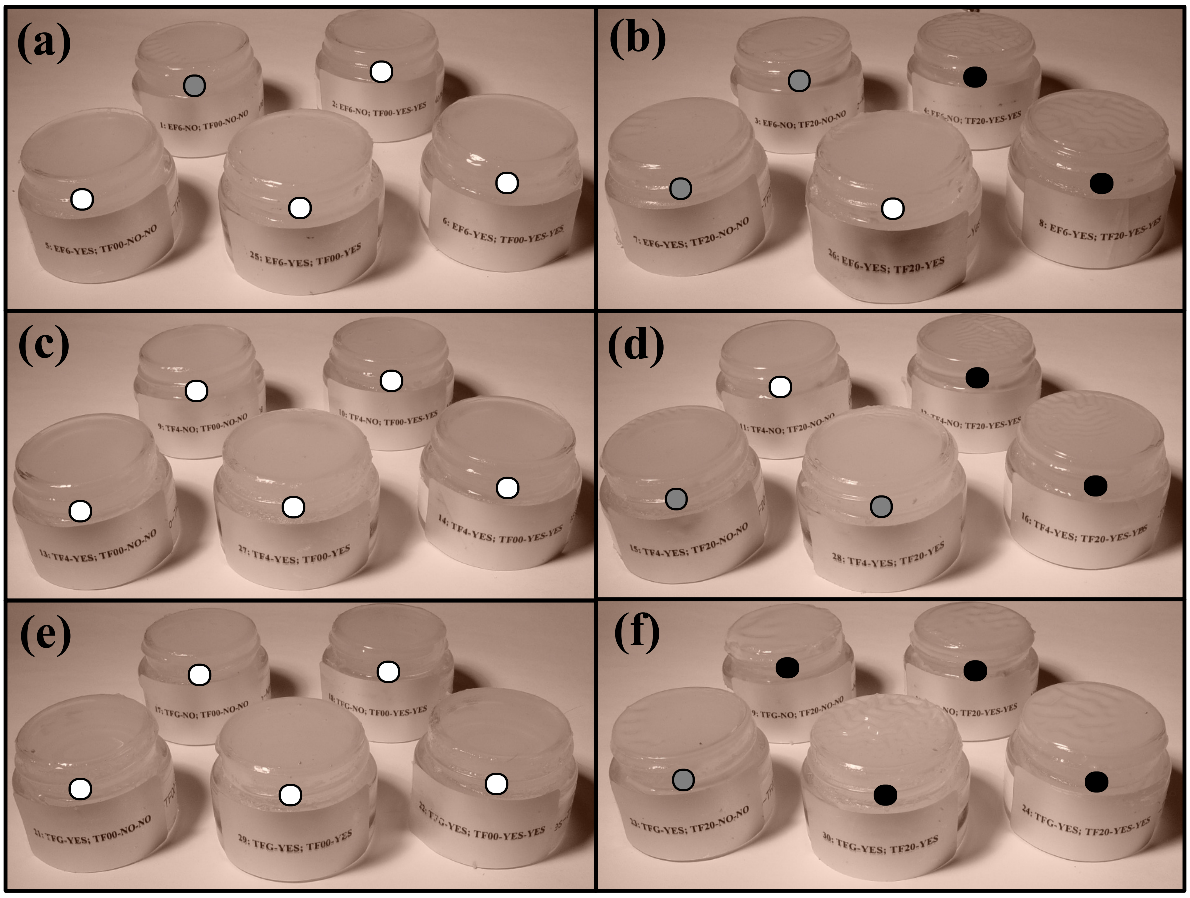

Figure 3 – Waviness of tempered silicone samples – (a) Samples of EF6 basis matter and TF00 epithelial layer; (b) Samples of EF6 basis matter and TF20 epithelial layer; (c) Samples of TF4 basis matter and TF00 epithelial layer; (d) Samples of TF4 basis matter and TF20 epithelial layer; (e) Samples of TFG basis matter and TF00 epithelial layer; (f) Samples of TFG basis matter and TF20 epithelial layer; – white circle: smooth surface; gray circle: slightly deformed surface; black circle: strongly deformed surface;

3.3 Discussion

The tests have shown that the production of synthetic vocal folds is best achieved by using the methods for the production of models 02, 48 and 49. A powder layer results in acceptable improvements even without additional tempering. A combination of both tempering and powdering could be the basis for further experiments. The talcum powder has slightly better values than [Ab] and the advantage of better process ability and availability. However, the threshold value, i.e., the weight and number of stick events, at which the stickiness is sufficiently low to ensure natural oscillation of synthetic vocal folds is still open. This has to be investigated by further studies in which vocal folds are produced using the described methods and then analyzed with respect to the vibration behaviour at different pressures. In addition, the measuring method shows considerable fluctuations in the times during which the weights stick to the samples, which is why it is reasonable to verify the results by producing further samples using the described manufacturing methods. Nevertheless, differences could be observed and a pre-selection of processes and materials for the production of synthetic vocal folds could be made.

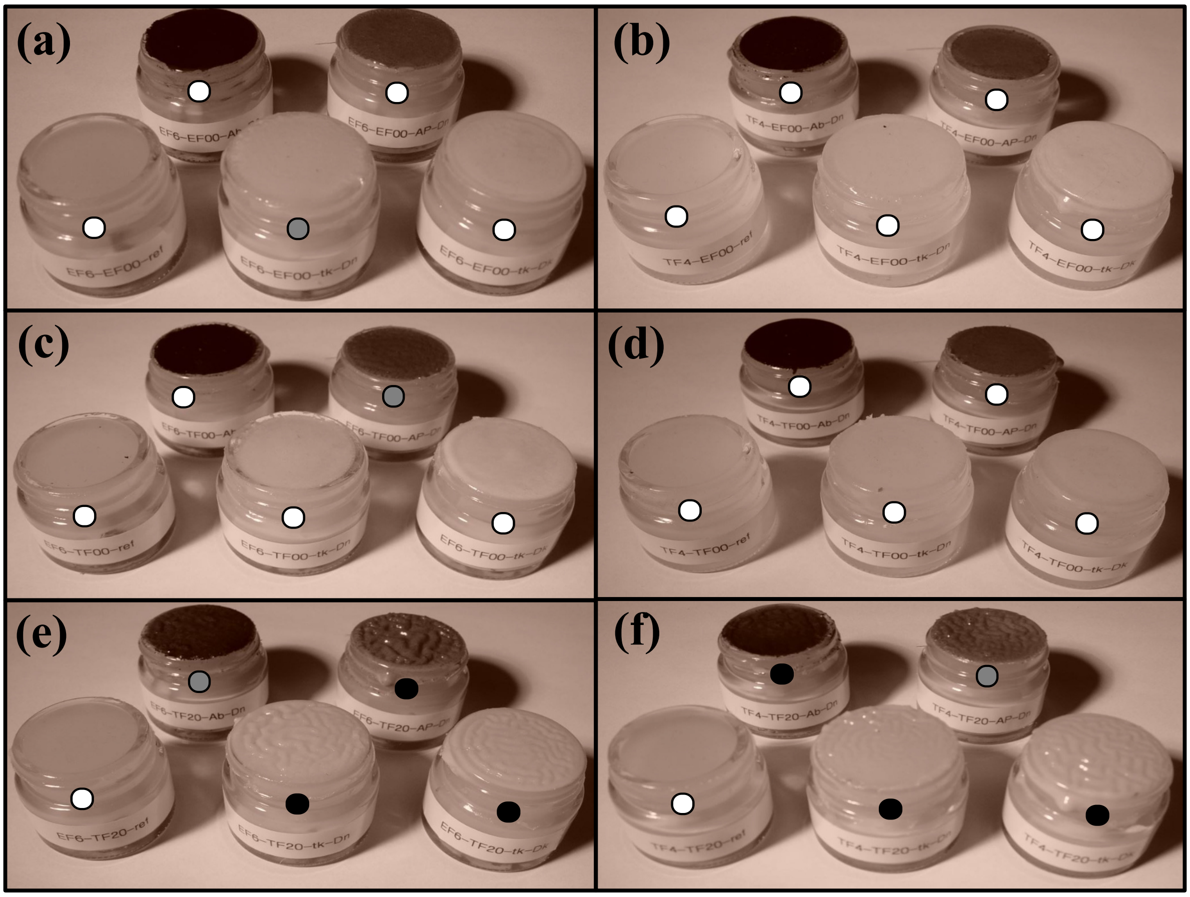

Figure 4 – Waviness of silicone samples with powder layer – (a) Samples of EF6 basis matter and EF00 epithelial layer; (b) Samples out of EF6 basis matter and TF00 epithelial layer; (c) Samples of EF6 basis matter and TF20 epithelial layer; (d) Samples of TF4 basis matter and EF00 epithelial layer; (e) Samples of TF4 basis matter and TF00 epithelial layer; (f) Samples of TF4 basis matter and TF20 epithelial layer; – white circle: smooth surface; gray circle: Slightly deformed surface; black circle: Strongly deformed surface;

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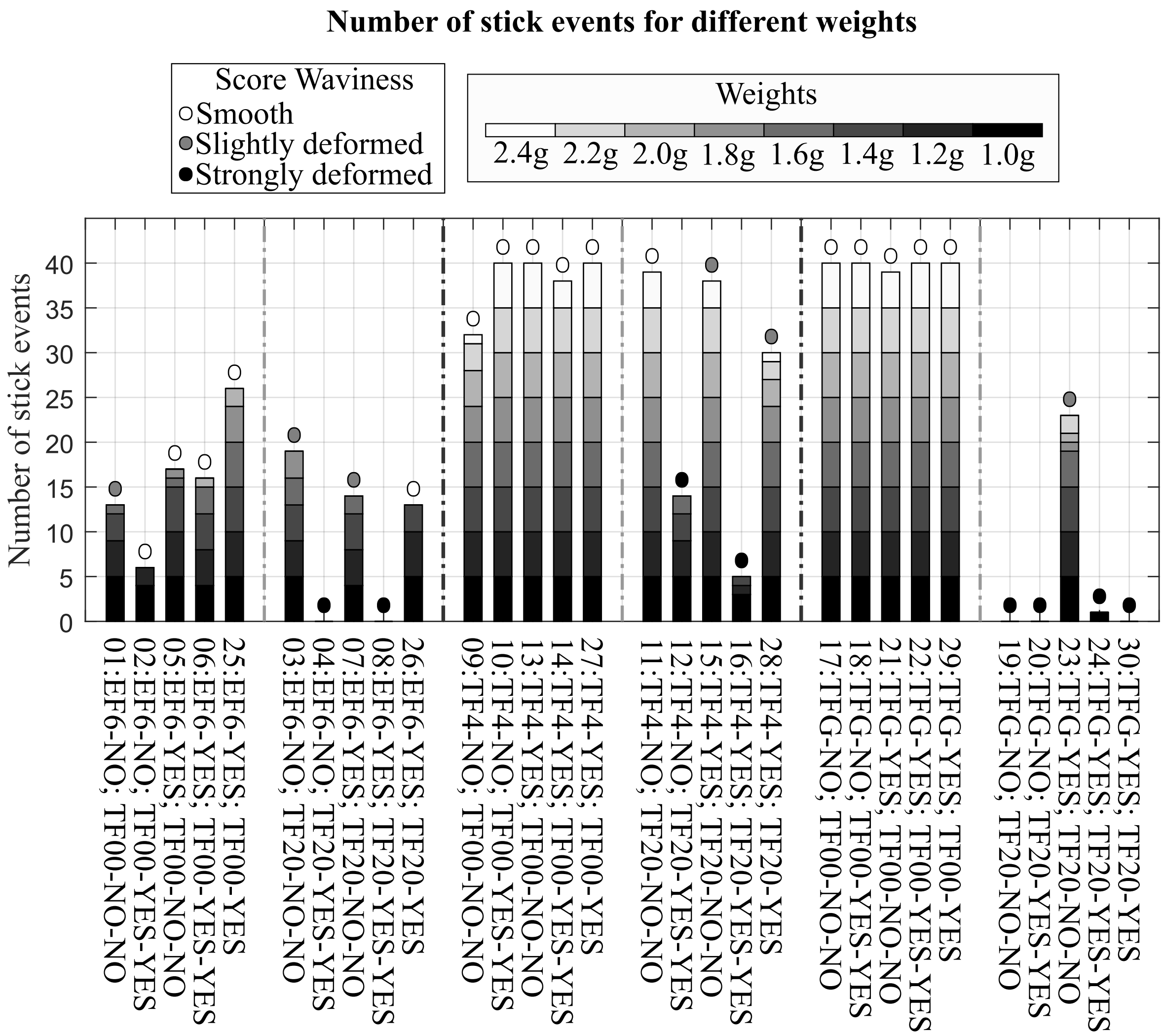

Figure 5 – Stickiness of tempered silicone samples; Number of stick events of each weight.

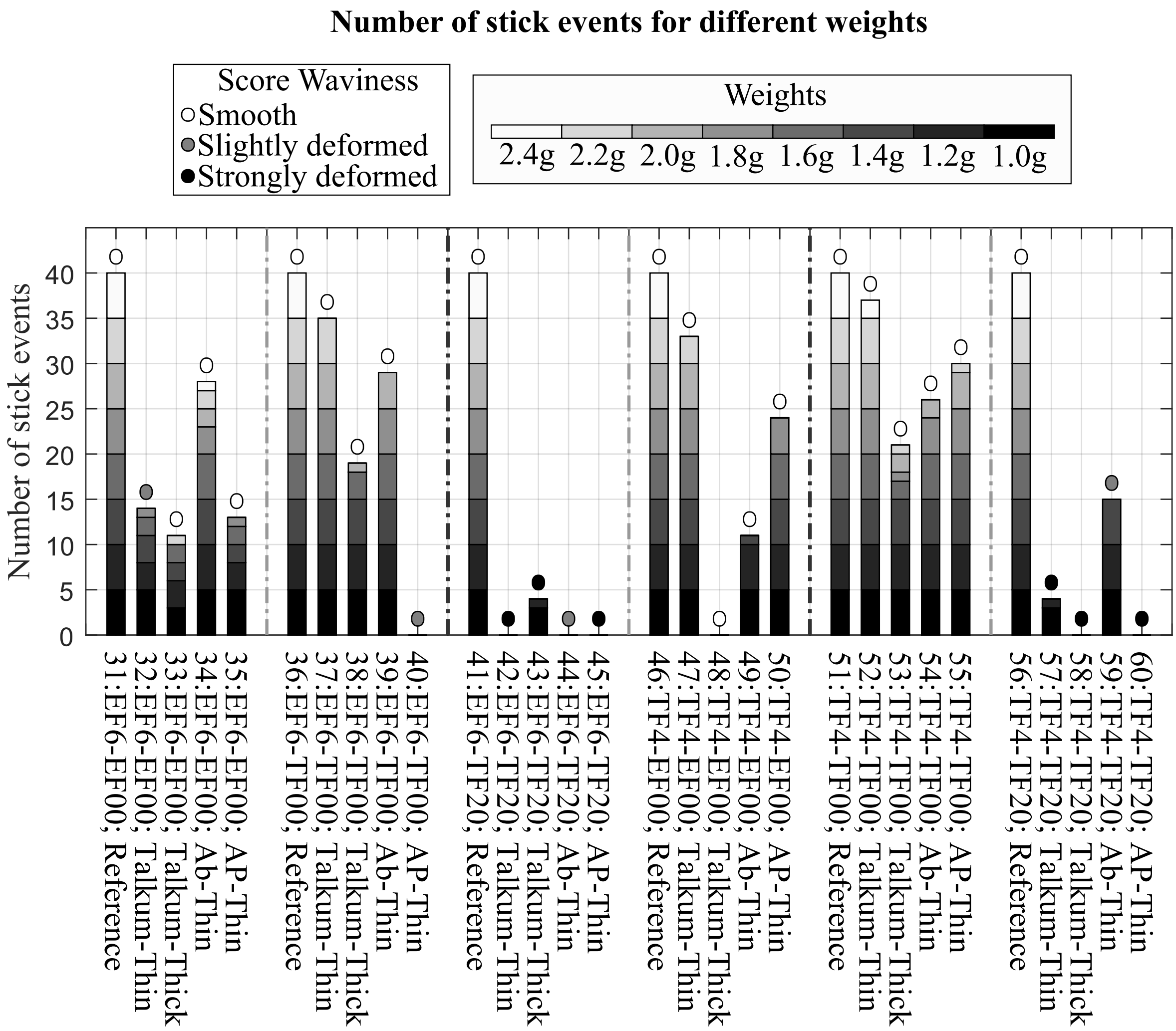

Figure 6 – Stickiness of silicone samples with powder layer; Number of stick events of each weight.