Vibration damping properties of steel/rubber/composite hybrid structures

Citation

Year
2012

Version
Peer reviewed version (post-print)

Link to publication
TUTCRIS Portal (http://www.tut.fi/tutcris)

Published in
Composite Structures

DOI
10.1016/j.compstruct.2012.04.035

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Abstract

By using hybrid structures, attractive and advantageous combinations of material properties can be achieved. In addition to the combination of good mechanical properties and low weight, also dynamical properties can be enhanced by suitable materials selection. However, the hybrid structure properties depend on the properties of the constituent materials as well as on the interfacial properties.

In the present study, the damping properties of laminated structures consisting of steel, rubber or epoxy adhesive and glass fibre reinforced epoxy composite were studied. Damping properties of the structures were investigated through the loss factors. The loss factors of the hybrid structures and the constituent materials were determined by frequency and time domain test methods. By using the loss factor results of the constituent materials, the loss factor of the hybrid structures were estimated by the rule of mixtures and the results were compared with the experimental results. It was observed, that the use of weight fractions instead of volume fractions in the rule of mixtures provides a good average estimation of the damping behaviour of the hybrid structure and the results of rule of mixtures method can be used as rough estimates during the design phase of hybrids of this kind.

Keywords: Hybrid structures, Vibration damping, Loss factor, Rule of mixtures

1. Introduction

Hybrid structures enable the utilization of the most advantageous material properties of different material grades and incorporation of them into one structure. A typical desired material property combination of hybrid structures is low weight combined with good mechanical properties. The number of beneficial property combinations is numerous, since the constituent materials of a hybrid structure may have substantially different material properties.

The manufacturing stage of a hybrid structure is usually challenging. Especially in laminated hybrid structures, in which macroscopic mechanical interlocking between the layers is unfeasible, the attainable adhesion level between the constituent materials may be insufficient. Since the interface quality often determines the service performance of the structure, additional surface treatments or adhesion layers are generally applied in hybrid laminates. Within steel/polymer hybrids, chemical surface treatments such as etching and anodizing [1-3], or additional interface layers such as coupling agents, adhesives, or coatings [2-4] are used to achieve good adhesion level. In addition, surface roughness of steel is often increased by grit blasting to enhance the interfacial strength. The use of chemical surface treatments usually highlights the issue of noxious chemicals and thus the use of additional interface layer may be favoured. However, the
drawbacks of the surface treatments are increasing manufacturing time and costs and thus it would be beneficial if they would not be needed.

Rubbers can be modified with additives to achieve good bond strength with steel as well as with polymers without surface pre-treatment [5-6] and thus it is a potential adhesive material. In addition to good adhesion properties, the viscoelastic nature of rubbers can be utilized in hybrid structures: the elasticity can equalize the internal stress concentrations at the interfaces and the good energy absorption properties can improve the dynamic properties of the structure. Further, the rubber can be vulcanized directly between steel and composite layers, which ensures a simple manufacturing process. However, in spite of its potential, rubber has not yet been widely used as adhesive in metal/polymer hybrids.

In an engineering system, a structure should stay stable and undamaged despite of the internal and external vibrations. The stability of a system depends on its damping ability, which can be affected by design. Damping is defined as the energy dissipation of a system in vibration and it can be presented by various parameters, one of which is the loss factor. Loss factor $\eta$ is defined as the specific damping capacity $D$ per radian of the damping cycle [7]:

$$\eta = \frac{D}{2\pi} = \frac{\Delta U}{2\pi U_{\text{max}}}$$

(1)

where $\Delta U$ is the energy loss per cycle and $U_{\text{max}}$ is the total stored reversible energy of the system. Parameters $\Delta U$ and $U_{\text{max}}$ can be measured through the area and the shape of the hysteresis loop in a stress-strain diagram of the damping cycle. The loss factor corresponds to the phase angle between strain and stress ($\tan \delta$) in Dynamical Mechanical Analysis (DMA):

$$\eta = \tan \delta.$$  

(2)

In addition, the loss factor can be defined as the ratio of loss and storage moduli:

$$\eta = \frac{E''}{E'}.$$  

(3)

Loss factor can be determined by several different methods, which are divided in two categories: frequency domain and time domain tests. Examples of the frequency domain methods are the half-power point and the magnification-factor methods, and examples of the time domain methods are logarithmic decrement and hysteresis loop methods [7-8].

The estimation of the dynamical properties with an effective method would be beneficial during the design phase of a hybrid structure. Since 1970’s, there has been an attempt to derive an expression for the loss factor of a reinforced composite [9], but it has been found to be highly sensitive to fibre architecture [10-11]. Instead, as a simpler approach to estimate the dynamic properties of composites, rule of mixtures could be used. Hashemi et al. [12] have studied the impact strength values of glass fibre/glass bead reinforced hybrid composites and found a positive deviation for the rule of mixtures behaviour when compared to the experimental data. However, if research is focused on hybrid structures where composite is one of the constituents, the composite component can be represented with homogenous equivalent component and the rule of
mixtures can be applied for the hybrid structure. For example, Botelho et al. [13] have used the rule of mixtures for aluminium based fibre metal laminates and found, similarly to the Hashemis results, a positive deviation for the estimated loss factor values when compared to experimental results. However, in [13], the effect of the aluminium/composite interface was not taken into account.

In the present study, the damping properties of hybrid structures based on alloy steel sheets and glass fibre reinforced plastic composite layers were studied. Both cold rolled and stainless steel grades were used. A thin EPDM based rubber layer or a commercial epoxy adhesive was used as an adhesive leading to four different hybrid structure types. The loss factors of the hybrid structures and the constituent materials were determined by two methods, based on frequency and time domain testing. The loss factor of the plain rubber was determined with dynamical mechanical analysis. By using the loss factor results of the constituent materials, the loss factor of the hybrid structures were estimated by the rule of mixtures and the results were compared with the experimental results.

2. Materials and methods

2.1 The samples

In this study, laminated structures of steel and glass fibre reinforced plastic (GFRP) composites were studied. As an adhesive between the steel and GFRP layers, an EPDM based rubber or an epoxy adhesive was used. Two kinds of steels were used: cold rolled steel EN 10130 DC01 (Ruukki Oy, Finland) and stainless steel AISI 304 (Outokumpu Stainless Oy, Finland). The surface of the cold rolled steel has been passivation treated and the stainless steel surface finish is one of the most widely used grade 2B (cold rolled, heat treated, pickled and skin passed). The GFRP layers were manufactured by vacuum infusion from 0/90 E-glass fibre mats (682 g/m²) and Sicomin SR 1660 / SD 7820 epoxy. The fibre content of the GFRP was 46%. The heat resistant epoxy was chosen to provide the resistance of the GFRP sheet to the vulcanizing temperature of the rubber. The rubbers were manufactured by Kraiburg GmbH, Germany and two different rubber grades were used to provide good adhesion with the different steel grades. The rubbers were particularly designed to create a strong adhesion between the metals and the GFRP layers used in this study. Especially the rubber grade which is compatible with stainless steel generates novel possibilities to utilize steel/rubber hybrid structures. The adhesive used was 3M™ Scotch-Weld™ Epoxy Adhesive DP190 Gray which exhibits good peel, shear and environmental aging properties.

The steel/rubber/GFRP structures were manufactured by vulcanizing the rubber between metal and GFRP layers under heat and pressure (@130°C, 1.2 MPa). Thin wires were used between the steel and GFRP sheets during the manufacturing step to provide uniform rubber thicknesses. The structures bonded with adhesive were cured in compression at room temperature. Plain steel and GFRP plates were used as reference samples. Table 1 lists the samples used in the damping tests. An example of the cross sectional samples are shown in Figure 1.

The thicknesses of the hybrid structure layers were studied from cross-sectional samples with optical stereomicroscope Leica MZ 7.5. Wallace X21B Electronic Densimeter was used to measure the densities of the different rubber grades and the GFRP plate.
The quality of the hybrid structures and especially their interfaces were investigated from the sample cross-sections with Scanning Electron Microscope (SEM) Zeiss ULTRAtplus. Conventional metallographic cross-sectional sample preparation method, including cutting the sample from the original damping specimens, mounting to epoxy, grinding, and polishing, was used to prepare the cross-sectional specimens.

Dynamic mechanical analysis (DMA) was carried out for both rubber grades to measure the loss factor of the plain rubber. The DMA tests were performed in shear mode with DMA/SDTA861 from Mettler at 22°C. The frequency range of the equipment is from 1 Hz to 1000 Hz, but the loss factor could be measured only up to 100 Hz due to the properties of the rubbers.

Two different test methods, namely frequency and time domain tests, were used to define the damping properties of the hybrid structures and the plain composite and steel sheets. In both tests the vibration is forced and the displacement of the sample is measured with a laser. Both methods were carried out at room temperature. The methods are described in the following two subchapters.

2.2 The frequency domain test

The frequency domain test used in this study was a free hanging plate test and the loss factor calculations were based on the fractional power bandwidth method (FPBW) [14]. In this method a plate sample (200x200mm) is suspended with two elastic wires and excited with a shaker. The shaker is stud-mounted to the specimen and the suspension generates free-free boundary conditions, which is generally accepted to be the only one suitable for a complicated structure [15]. The displacement of the plate during excitation is detected with a laser at certain measurement points. The test setup is illustrated in Figure 2 and a more specific description of the test setup can be found in [16]. A similar test setup has been used, e.g., by Matter et al. [17].

The free hanging plate test method enables the evaluation of loss factor only in resonance, i.e., at discrete eigenmode-points starting from the first resonance frequency of the sample. To include the first resonance frequency to the tested frequency scale, the first resonance frequency was determined for each sample individually before the actual damping test. Then the specimens were scanned in a frequency scale up to 500 Hz with a sine-sweep signal from 25 measurement spots and the frequency response function (FRF) of the structure’s mobility was recorded. The locations of the measurement spots and the excitation point are shown in Figure 2. The reflectivity of the measurement spots were enhanced by using a reflective tape.

To determine the behaviour of the whole plate, a mean frequency response function was calculated with a root mean square of the FRFs of the individual measurement points:

\[
FRF_{plate\ mobility} = 20 \cdot \log \left( \sqrt{\frac{\sum_{i=1}^{m} |v_i|^2}{m}} \right)
\]

where \(m\) refers to the number of measurement points (\(m=25\)), \(v\) is the velocity of the studied point and \(F\) is the input force of the excitation. In the mean FRF, each peak represents a resonance frequency of the plate and the modal loss factor \(\eta\) can be calculated from the peaks with the fractional power bandwidth method:
\[ \eta = \frac{1}{\sqrt{10^{10} - 1}} \frac{\Delta \omega}{\omega_{res}} \]  
\[ (5) \]

where \( n \) is the amplitude decay of \( n \) dB in frequency domain, \( \Delta \omega \) is the frequency range of decay in radian and \( \omega_{res} \) is the resonance frequency in radian. The standard [14] defines the \( n \) to be between 0.5 and 3 dB.

The free hanging plate test method is accurate only for structures with small damping values (loss factor up to 0.2-0.3) since in the FRF some peaks will be eliminated by remarkable damping [8]. In addition, the damping difference predicted by various theories becomes more pronounced with increasing damping.

2.3 The time domain test

The time domain test method is based on Dynamic Mechanical Analysis (DMA). In this method, a beam sample (40x200mm) is vibrated in three-point bending configuration. The displacement of the beam during excitation is detected with a laser from the centre of the specimen. The test setup is illustrated in Figure 3 and a more detailed description of the test setup can be found in [18].

This test method enables testing in a continuous frequency range below the first resonance frequency of the sample which in current case located around 60-170 Hz. In this study, the testing was started from 20 Hz and the step size used was 30 Hz.

The phase shift discussed in Equation 2 is between the force \((\varphi_F)\) and the displacement \((\varphi_x)\) data as described in the following equation:

\[ \eta = \tan(\varphi_x - \varphi_F). \]
\[ (6) \]

The phase shift is evaluated directly from the output data. Thus the DMA based time domain test is more reliable if the loss factor of the investigated structure is high when the phase shift is distinct and evaluation errors are small.

When there was a slight phase shift between the force curves detected from both ends of the sample, the force curve with the smaller phase shift compared to the displacement curve was used in the loss factor evaluation. The phase shift was assumed to exist due to the twisting of the samples. An average of five phase shifts was calculated for each frequency.

3. Results

3.1 The density measurement results

The measured densities were 1760 kg/m\(^3\) for the GFRP plate, 1110 kg/m\(^3\) for the rubber grade A and 1130 kg/m\(^3\) for the rubber grade B. The density is 7850 kg/m\(^3\) for the cold rolled steel [19] and 7900 kg/m\(^3\) for the stainless steel [20]. According to the volume fractions of the constituent materials, the weight fractions of the components were calculated, as shown in Table 2.

3.2 The microscopy results
The nominal layer thicknesses studied with the optical microscope are presented in Table 1. The SEM studies revealed a close contact between the constituent materials in all hybrid structures. The GFRP surface had undulating and coarse contour and thus provided mechanical interlocking for the rubbers and epoxy adhesive. On the contrary, the steel surfaces were flat and smooth and mechanical interlocking at the steel surfaces was minimal. No difference was observed in the quality of the GFRP/rubber A, the GFRP/rubber B, or the GFRP/epoxy adhesive interfaces. Likewise, the cold rolled steel/rubber A, the stainless steel/rubber B, and steel/epoxy adhesive interfaces were similar. Examples of the SEM images taken from the samples are shown in Figure 4.

### 3.3 The loss factor studies

The average response curves of the frequency domain test were calculated for all of the specimens in Table 1. Two examples are presented in Figure 5. A shift of certain resonance frequencies towards higher frequencies can be seen in the sample with higher damping capacity, i.e., thicker rubber layer.

The loss factor results from the frequency domain tests are shown for the plain steel and GFRP plates (samples 9-11) in Figure 6 and for the epoxy adhesive hybrids (samples 7-8) in Figure 7. The loss factor result values for the rubber hybrids (samples 1-6) are shown in Figure 10.

The average loss factors from the time domain tests are shown in Figure 8 and the DMA results for rubber A and B are shown in Figure 9.

### 4. Discussion

The loss factor of the plain cold rolled steel sheet and bare stainless steel sheets from the frequency domain tests (Figure 6) are around 0.005-0.01. The loss factor of steel given in literature is $2 \times 10^{-5}$ [8, 21-22], so it can be assumed that in the frequency domain test setup a slight positive systematic error exists. The stud-mounting of the vibrator and the suspension are examples of the systematic error sources. A general trend of higher loss factor values at lower frequencies can be seen in the results.

According to the frequency domain tests, the damping properties of the GFRP plate (Figure 6) appeared to be remarkably higher when compared to the metal sheets, which is expected since the more effective and versatile damping mechanisms are included in fibre reinforced composites when compared to the metal sheet. The damping mechanisms in GFRP, in addition to the viscoelastic nature of the GFRP’s constituents, are damping due to the fibre-matrix interface, thermoelastic damping due to the heat flow and damping due to damage [23]. Damping in the metal sheet is due to the effect of microdefects, heat formation, corrosion, and damage initiation [13]. In addition to higher loss factor of GFRP, the frequency dependence of the GFRP’s loss factor is stronger when compared to the steels.

The loss factor values of cold rolled steel based hybrids (samples 1-3) were somewhat lower than the loss factors of the stainless steel based hybrids (samples 4-5) as illustrated in Figure 10. Since there was no remarkable difference in the loss factor values of the cold rolled and stainless steel plates and as the GFRP sheets are similar in all the samples, the difference must arise from the higher loss factor of rubber B (Figure 9). In addition, especially in the results of stainless steel based rubber hybrids an
increase in the loss factor with increasing rubber thickness can be seen. The frequency
dependence of GFRP plates can also be seen in the hybrid structures.

The loss factors of the epoxy adhesive hybrids (samples 7 and 8, Figure 7) were similar
to the steel/rubber hybrids (samples 1-6). This suggests that the damping effect of the
GFRP component is more dominant than the effect of the thin rubber layer.

The loss factor results of the time domain test were generally higher than the results of
the frequency domain test. However, scattering of the time domain test results was
stronger when compared to the frequency domain results and such trends which were
seen in the frequency domain test results, were lacking from the time domain test
results. Thus, the frequency domain test was more suitable for the studied hybrids.

In this study, the rule of mixtures (ROM) was employed for the frequency domain test
results to investigate, whether the ROM method would be applicable for this kind of
hybrid structures. The results from the frequency domain test for the plain steel and
GFRP sheets and the DMA values for the rubbers were used as starting values. It was
assumed, that the saturation of the DMA results of the rubber grades would keep a
similar loss factor until 500 Hz. Both weight fractions and volume fractions were used
to weight the loss factor values of the individual components. The ROM results for the
steel/rubber/GFRP hybrids are depicted in Figure 10 together with the experimental data
and the root mean square deviation (RMSD) values for the weight and volume fractions
ROM estimations when compared to the experimental data are shown in Table 3.

It can be seen from Figure 10 and from Table 3, that the use of volume fraction
overestimates the loss factor results. Instead, the ROM results based on the use of the
weight fractions seem to cohere better with the experimental results. The better
suitability of the weight fraction instead of the volume fraction in the application of
ROM, is supposed to be due to the dominating nature of the steel sheets in hybrid
structures. When volume fractions are used in ROM, the role of GFRP and rubber is
emphasized and if weight fractions are used, the role of steel sheet becomes more
important, as can be seen from Table 2. However, steel is much stiffer than GFRP or
rubber, so it dominates the deformations of the hybrid structure and perhaps diminishes
vibrations and damping in the other components. In addition, the energy lost during
deformation is the product of the loss factor and modulus of the material (Equation 3),
which increases the importance of the steel sheet further.

At certain measurement points, there is a noticeable positive or negative deviation in the
experimental results from the ROM behaviour. Thus, only the average loss factor of the
steel/rubber/GFRP hybrids can be estimated by the ROM. The effect of the interfacial
damping mechanisms has to be small when compared to the damping capacity of the
constituent materials, since the average loss factor values fitted such well with the
experimental data.

The ROM results seem to reproduce loosely the trends of the experimental results.
Because the behaviour of the metal sheets and the rubber seem not to be strongly
frequency dependent within the measurement range, the similarity between the
experimental data and the ROM estimation consolidates the assumption that the strong
frequency dependence of the hybrids are rather due to the systematic effect of the GFRP
behaviour than measuring error. In addition, the effect of the GFRP component in ROM
estimation may be too low, as the ROM results are less frequency dependent than in the
experimental data.
5. Conclusions

Damping properties of hybrid structures consisting of a metal sheet, adhesive and composite sheet were investigated. Cold rolled low alloy steel and stainless steel were used as metal components and glass fibre reinforced epoxy composite was used as the composite layer. Two different rubbers and an epoxy adhesive were used between the metal sheets and the composite. The loss factors of the individual components and of the hybrid structures were studied with two methods, based on frequency and time domain testing. The former method proved to assort to the tested materials better.

Unlike the damping capacity of steel and rubber components, the damping capacity of GFRP proved to be strongly frequency dependent. The frequency dependence was also observable in the case of the hybrids, as well. Especially one can notice the better shape of frequency versus damping matching of weight fraction ROM when compared to volume fraction ROM. In addition, increasing rubber thickness increased the loss factor of the structure. Thus, to increase the damping capacity of the hybrid structure, the rubber layer thickness should be increased. In such case it should be kept in mind that the mechanical durability of the thick rubber layer can turn out to be insufficient.

Rule of mixtures was applied for the frequency domain test results. When the loss factor values of individual components were weighted according to the weight fractions, a good estimation of the average loss factor of the hybrid structure was achieved. In addition, the estimated loss factor reproduced the trends of the experimental frequency dependence of the loss factor. Thus the loss factor of this kind of three layer hybrids can be estimated by the rule of mixtures during the design phase. However, to improve the accuracy of the estimation, the effect of GFRP component should be emphasised.

Acknowledgements

The work was founded by the Doctoral Programme of TUT’s President. The authors acknowledge Outokumpu Oyj for the stainless steel sheets, Rautaruukki Oyj for the cold rolled steel sheets and Kraiburg GmBh for the rubbers. In addition, the authors are thankful for Kosti Rämö from Tampere University of Technology for the manufacturing of the GFRP composites and hybrid structures used in this work and for the density measurements.

References


Table 1: The samples used in the damping tests and the nominal thicknesses of the components.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Metal plate</th>
<th>Adherent</th>
<th>GFRP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cold rolled steel 0.5 mm</td>
<td>EPDM grade A 0.5 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>2</td>
<td>Cold rolled steel 0.5 mm</td>
<td>EPDM grade A 1.0 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>3</td>
<td>Cold rolled steel 0.5 mm</td>
<td>EPDM grade A 1.5 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>4</td>
<td>Stainless steel 0.5 mm</td>
<td>EPDM grade B 0.5 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>5</td>
<td>Stainless steel 0.5 mm</td>
<td>EPDM grade B 1.0 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>6</td>
<td>Stainless steel 0.5 mm</td>
<td>EPDM grade B 1.5 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>7</td>
<td>Cold rolled steel 0.5 mm</td>
<td>Epoxy adhesive 0.14 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>8</td>
<td>Stainless steel 0.5 mm</td>
<td>Epoxy adhesive 0.19 mm</td>
<td>GFRP 1.3 mm</td>
</tr>
<tr>
<td>9</td>
<td>Cold rolled steel 0.5 mm</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>Stainless steel 0.5 mm</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>11</td>
<td>-</td>
<td>-</td>
<td>GFRP 1.3 mm</td>
</tr>
</tbody>
</table>

Figure 1: Cross-sections of a) the stainless steel/rubber/GFRP hybrid and b) the stainless steel/epoxy adhesive/GFRP hybrid.
Figure 2: The frequency domain test setup and the measurement, supporting and excitation points in the samples.

Figure 3: The time domain test setup [19].
Table 2: The volume and weight fractions of the components in different rubber hybrid structures.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Volume fractions [%]</th>
<th>Weight fractions [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Metal</td>
<td>Rubber</td>
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<tr>
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<td>0.28</td>
</tr>
<tr>
<td>2</td>
<td>0.21</td>
<td>0.34</td>
</tr>
<tr>
<td>3</td>
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<td>0.47</td>
</tr>
<tr>
<td>4</td>
<td>0.17</td>
<td>0.37</td>
</tr>
<tr>
<td>5</td>
<td>0.17</td>
<td>0.39</td>
</tr>
<tr>
<td>6</td>
<td>0.15</td>
<td>0.46</td>
</tr>
</tbody>
</table>

Figure 4: SEM images of the interfaces of a) stainless steel and rubber B, b) GFRP and rubber A, c) cold rolled steel and epoxy adhesive, and d) GFRP and epoxy adhesive.

Figure 5: Examples of the average response curves of the frequency domain test. Sample 4 corresponds to the stainless steel/rubber 0.5 mm/GFRP hybrid and Sample 5 corresponds to the stainless steel/rubber 1.0 mm/GFRP hybrid.
Figure 6: The loss factor results of the plain a) cold rolled steel (sample 9), b) stainless steel (sample 10), and c) GFRP plates (sample 11) in frequency domain test.

Figure 7: The loss factor results for the epoxy adhesive hybrids a) sample 7, and b) sample 8 in frequency domain test.

The average loss factors from the time domain tests are shown in Figure 8 and the DMA results for rubber A and B are shown in Figure 9.
Figure 8: Average time domain test results.

Figure 9: The DMA results for rubber A and B.
Figure 10: The frequency domain test results (blue), the weight fraction ROM results (green), and the volume fraction ROM results (red) for the cold rolled steel/rubber hybrids: a) sample 1, b) sample 2, c) sample 3, and for the stainless steel/rubber hybrids: d) sample 4, e) sample 5, f) sample 6.
Table 3: The root mean square deviation (RMSD) values for the weight and volume fractions ROM estimations when compared to the experimental data for the cold rolled steel/rubber hybrids (samples 1-3) and for the stainless steel/rubber hybrids (samples 4-6).

<table>
<thead>
<tr>
<th>Sample</th>
<th>RMSD for weight fraction ROM estimation</th>
<th>RMSD for volume fraction ROM estimation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0087</td>
<td>0.0132</td>
</tr>
<tr>
<td>2</td>
<td>0.0120</td>
<td>0.0186</td>
</tr>
<tr>
<td>3</td>
<td>0.0089</td>
<td>0.0198</td>
</tr>
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<td>4</td>
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<td>0.0180</td>
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<td>0.0148</td>
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<tr>
<td>6</td>
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