A NOVEL COMPOSITION FOR REMOVABLE INTERNAL TOOLING OF HOLLOW COMPOSITE STRUCTURES

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Keywords: mandrel, internal tooling, hollow parts, RTM, composite manufacturing, composite processing

1 Introduction

The importance of lightweight design in many industries is growing, leading to higher demands for fiber reinforced plastic (FRP) products. FRP components combine high mechanical properties with low mass.

The bending stiffness of a component increases with increasing distance of the component surface to the neutral axis (i.e. with increasing thickness for monolithic structures). Different loads continue through the outer area and the inner area. Thus components are often manufactured from two different materials; a stiff material for the surface “skins” and a low density material for the inner core. Removing the inner material reduces the weight of the component at the same stiffness and results in a hollow component. The manufacturing process of hollow composite components can be distinguished as differential and integral design. In differential design, components are assembled from several pre-manufactured elements, whereas in integral design the component is produced in a single manufacturing process. The production of integral hollow metallic components is a well-known manufacturing process. The integral design promises lower assembly costs, less weight and structural advantages due to continuous load carrying fibers in the component. However, the complexity of the manufacturing process grows due to the need for additional tooling for the hollow space. These internal tools for FRP must be removable, to maximize the lightweight potential of the component.

In Figure 1 the basic concept of internal tooling, also known as mandrel or core, with and without undercuts and different core materials is depicted.

A number of mandrel systems have been investigated for industrial applications, in an attempt to introduce the technology to series production. In the automotive sector a carbon fiber reinforced plastic (CFRP) bumper with a sand core was produced [1]. Furthermore blow-molded thermoplastic, multi-part metal and cast wax cores for the production of FRP components are used. For an aircraft passenger door CFRP hinge arms are produced with water soluble cores [2]. In the manufacturing of composite helicopter structures the use of flexible tubes is common as well as in the production of bicycles and rackets. However, the majority of these core materials result in poor surface quality. For most of these components the outer surface has a high surface quality due to the use of a rigid outer tool. The inner surface quality is not considered during the manufacturing process, so time- and cost-intensive rework is required to achieve a high quality surface finish of the inner surface.

The general requirements for core materials are derived from process and component specifications. The general requirements for a core material are:

- dimensional stability,
- compressive and flexural stiffness,
- removability,
- resin impermeability,
- temperature resistance,
- surface quality,
- costs.

The challenge in developing a new core material is to meet the costs of existing core material systems and provide at least the same properties regarding the flexural stiffness, compressive stiffness, dimensional stability and removability.

The removability of an inner tooling is of particular importance. Full removal of the core material from an undercut FRP component is essential to gain the complete lightweight potential. The removal must be
a process in which the composite part is not damaged, reducing the mechanical properties.

The surface quality is of particular relevance. The majority of the current inner tooling systems are not able to achieve the required surface roughness. Therefore a mean roughness value $R_s$ of at least 5µm is targeted.

Resin systems used for the manufacturing of composites in aerospace industry often require a heat treatment above room temperature. This is necessary to achieve the operating viscosity and/or to cure the resin completely. Typical matrix systems are Hexcel’s RTM6 or CYCOM 977-2 from Cytec. The realizable temperature is derived from the process cycles of the above mentioned resin systems and set at 200°C.

In this paper a novel internal tooling method, using a multi-material approach, is presented. Different materials are used to provide the mechanical properties of the internal tooling required for a robust preforming and saturation process; allow removal of the core via a change of state, and post curing the matrix.

2 Manufacturing of hollow composite components using removable inner tooling

The removable inner tooling defines the geometry of the hollow space of the future part with undercuts. The surface quality of the composite is a possible distinguishing feature of the tooling. By choosing the respective tooling either the surface quality of the inner or outer face, or both, can be adjusted. The required high surface quality can be achieved with a smooth removable lost core or a multi-part inner tooling. The term “lost core” describes a core which can be used once. Using a removable lost core, a multi-part inner tooling or a flexible hose in combination with a rigid outer tooling, a high surface quality of the outer surface is achievable. In order to realize high quality on both surfaces, a combination of core materials achieving high surface qualities is used: a rigid outer tooling and a removable inner tooling with smooth surfaces.

The core materials can be classified by the method of removal: changing the aggregate state (AS), destroying chemical bonds (CB) and extraction (EX). Examples of these three classifications are:

- **Meltable wax (AS):** Semi-crystalline polymer wax cores are produced using the casting method. Due to the self-releasing property of the wax no additional release agents are required. The shrinking of the polymer over several weeks leads to high variability in the geometrical tolerances. The mechanical stiffness and surface quality is sufficient for most applications. Common systems have a melting temperature of approx. 120°C.

- **Low Melting Alloy (AS):** Produced with the casting method the low melting alloy core is melted out of the composite. Sufficient stiffness, surface quality and impermeability are properties of the alloy. The temperature resistance is adjustable depending on the selected alloy: alloys with melting temperatures between 50°C and 200°C are available. The high specific density of the alloys and the toxic components (e.g. cadmium) of some alloys induce extra effort to ensure safety of handlers.

- **Water-soluble salt (CB):** Salt is processed to a defined geometry by casting or pressing. The pressing process of sodium chloride (NaCl) requires expensive and complex tools. The casting of the raw material is typically performed under pressure. Since the material is porous a sealant is required. The release of ammonia gas under temperature adversely affects the safety of handlers.

- **Fillers and water-soluble binder (CB):** The core is manufactured from a semi-finished product or directly in a pressing process near net-shape. Fillers such as sand, hollow ceramic or glass spheres can be used. The surface of the core requires sealing due to the porous structure.

- **Foams (CB):** The core can be foamed directly or processed from semi-finished product. The closed-cell foam is removable using solvents, although this is not eco-friendly. The mechanical properties are sufficient for the vacuum infusion processes; however, the compressive stiffness will not withstand processes with high injection pressures.

- **Metal (EX):** Metal-core tools typically consist of several parts in order to allow removability from the component. Temperature resistance, stiffness, dimensional stability, surface quality and resin impermeability are achieved with metal cores.

- **Thermoplastic (AS, EX):** Thermoplastic hollow cores are typically produced using the blow molding or rotational casting processes. The stiffness of the plastic itself is not sufficient for injection. By applying pressure with air or a liquid or by adding a solid filling material the stiffness can be increased. The temperature re-
3 Principle of multi-material system

Under a multi-material approach, the necessary different functions of the core material are assigned to different materials. One material provides, for example, the mechanical properties and the surface quality. Additives can be added to the material to adjust the properties if desired. The core material presented uses gypsum plaster as the main ingredient. Here, expandable graphite is added to ensure the complete removal of the core material. The expandable graphite begins expanding at a certain threshold temperature and the final expanded volume is a multiple of its initial volume. Due to this expansion the integrity of the plaster is damaged and disintegrates into small particles before the FRP is damaged. These particles and the expanded graphite can easily be removed from the composite component. The described processing is depicted in Figure 2.

The resin impermeability of the core material (described in Section 1) is required to enable removability of the core. For materials with high permeability, such as gypsum, an additional seal is required. The permeability is based on the materials porosity; resin seeps into the core material during infiltration. The core material bonds with the component internally and after curing a gentle removal is impossible. The requirements for the seal are:

- resin impermeability,
- temperature resistance,
- reproducibility and uniformity,
- geometrical tolerances,
- surface quality.

“Core material” is defined as the components which provide the geometry of the component, while the “inner tooling system” includes the core material and the applied sealing agent onto the core material.

3.1 Components of the multi-material system

In this section the different components of the core material are introduced. The core material comprises two different components: gypsum and expandable graphite. As gypsum is a porous material and with the addition of expandable graphite this property does not change a sealant is essential in order to prevent saturation and to allow removal of the core material. The sealant is considered an additional component of the inner tooling system as described above.

Similar material combinations are already used in other applications. For example expanded graphite is used to increase the thermal conductivity of plaster-based construction materials [9].

In this section gypsum, expandable graphite and sealing agents are introduced and raw material investigations are presented.

3.1.1 Gypsum

Gypsum is commonly known as a construction building material, as orthopedic casts or for use as a fertilizer. The chemical formula of this sulfate mineral is calcium sulfate dihydrate (CaSO₄·2H₂O). The chemically bonded water dehydrates partially as steam (Eqn. (1)) above temperatures of 150°C and completely above 180°C [5]. This reversible process is also called calcination and results in hemihydrate (CaSO₄·0.5H₂O) (Eqn. (1)) or anhydrate (CaSO₄) (Eqn. (2)). The rehydration of plaster to gypsum reacts exothermically after mixing with water. The expressions “gypsum” and “plaster” refer to dihydrate and hemihydrate, respectively.

\[
CaSO₄ \cdot 2H₂O \rightleftharpoons CaSO₄ \cdot 0.5H₂O + 1.5H₂O \quad (1)
\]

\[
CaSO₄ \cdot 2H₂O \rightleftharpoons CaSO₄ + 2H₂O \quad (2)
\]

Calcium sulfate is classified in five different phases, where the crystalline structure and the chemically bonded water differ:

- dihydrate (CaSO₄·2H₂O),
- hemihydrate (CaSO₄·0.5H₂O),
- anhydrate III (CaSO₄ III),
- anhydrate II (CaSO₄ II),
- anhydrate I (CaSO₄ I).
For the core materials used here, the first three phases are of interest due to the processing temperature range. These are depicted in Table 1. Hemihydrate occurs in α-hemihydrate and in β-hemihydrate. The anhydrate is hygroscopic and stabilization is critical. Water is absorbed from the ambient air and anhydrate alters to hemihydrate.

**Appearance of gypsum plaster**

In general gypsum plaster is provided in powder form. Different types of plaster are available, such as:

- modeling plaster,
- building plaster,
- alabaster plaster,
- dental plaster.

These types are composed of the two variants of hemihydrate and different additives. For the chemical reaction the plaster powder \( p \) is mixed with water \( w \) in a specific ratio \( r \). Information about the ratio is provided by the manufacturer:

\[
   r = \frac{w}{p} \tag{3}
\]

The ratio ranges from 0.28 to 0.4. For the investigation, commercially available modeling plaster produced by Meffert AG and a raw alpha hemihydrate produced by Knauf are used due to higher mechanical properties [3]. The modeling plaster is a mixture of alpha hemihydrate, beta hemihydrate and several unknown additives. These additives are a motivation to use raw alpha hemihydrate so that the mixture can be controlled. The cost of the raw alpha hemihydrate is 30% higher compared to the cost of the modeling plaster.

**3.1.2 Expandable graphite**

Expandable graphite is a special form of graphite in which additional molecules are intercalated. The intercalated molecules are primarily sulphur or nitrogen compounds. These intercalation compounds (GICs) or expandable graphite salts expand abruptly at a certain threshold temperature. Particular grades have threshold temperatures of 150°C up to 280-300°C. An expansion of 100 times the initial volume is possible during free expansion. The properties of the expandable graphite, such as threshold temperature and degree of expansion depend on the intercalation quality and the intercalated material.

Two materials ES200F5L (EG1) and GHLPX95LT (EG2) of different suppliers were used in previous trials which evaluate the expansion behavior. Both have a threshold temperature of 160°C.

**Expansion behavior**

In the technical data sheet of the expandable graphite the free expansion without any applied pressure is stated. In order to verify these values and to understand the behavior under pressure a Thermo Mechanical Analysis (TMA) was conducted. Typically the TMA is conducted in order to determine the coefficient of linear thermal expansion (CTE) of a solid sample. As it is not possible to provide expandable graphite in solid state the graphite is placed into a container. The displacement transducer is placed on the expandable graphite.

Three samples of each of the expandable graphite grade EG1 and EG2 have been tested. The stamp pressure of 0.05 N was applied to the sample while the temperature was raised from room temperature to 180°C. The resulting expansion for EG1 is 10 times the initial height and for EG2 is 17 times the initial height. In Figure 3 the expansion of EG1 and EG2 versus the temperature is presented. The expansion begins at 160°C and continues to a maximum at 200°C.

The expansion achieved at 200°C is at least 10 times the initial volume under a pressure of 0.05 N. Although this is not the process environment during the removal of a core from a CFRP component the high expansion potential can be demonstrated.

**3.1.3 Sealing agent**

The gypsum and the material composition presented here (gypsum combined with expandable graphite) are porous. In previous tests a saturation of the gypsum-based core system was detected. The cured resin in the core material prevents the removal of the core. Separation of the matrix material from the porous core material is necessary. Therefore a sealant is applied to the core which must satisfy all the requirements stated in the introduction. Most sealants offer a thermal stability up to 100-130°C. For the required temperature range no suitable ready-to-use solutions were available. The surface quality is not a requirement for the CFRP production in many applications; therefore the often-used process of wrapping the core with a plastic film is omitted in this investigation.

The evaluation of the sealants was performed on samples of modeling plaster. These samples had a simple tubular geometry. The following sealant materials were used:
• **PVA paint** is a commonly known sealing system which is typically used in combination with a wax. Although the paint has a maximum operating temperature of 100°C the PVA paint has previously been tested up to 150°C. Thus it was decided to test if this boundary could be stretched further.

• **Heat shrinkable tube** is generally used to isolate electrical conductors. The operating temperatures depend on the chosen plastic. Here, the chosen tube (Hi-Shrink PET Tubing) is sufficiently stable up to 180°C according to the manufacturer’s data sheet.

• **Micro-Isolat NF** is an alginic acid which is used to seal dental imprints. Applied to the surface of the gypsum, the alginic acid reacts with the calcium and develops a sealing layer. Temperatures above the maximum operating temperature of 100°C have never been tested as it is not required in dental medicine.

• **Temperature resistant paint 1 (TRP1)** is Bondupal R1012 with a temperature resistance up to 600°C. Paint can be applied to the core by brush or spray.

• **Temperature resistant paint 2 (TRP2)** is paint for heaters resistant up to 180°C. Paint application to the core is by brush.

• **Teflon tape** has been used for other CFRP parts as a sealant. The temperature resistance is up to 260°C. The tape is wrapped around the core with overlaps such that a tight surface is achieved. An additional release agent is not necessary due to the self-release properties of the Teflon.

For testing, the sealing agent was applied to the gypsum sample and then heated in an oven at 200°C for 4 hours (phase 1). If no obvious defects of the sealant occurred the cores were covered with two layers of braided textile and infused with resin by the Vacuum Assisted Resin Infusion (VARI) process (phase 2). The resin was cured at 180°C for 2 hours.

During the first evaluation phase the PVA paint and both temperature resistant paints showed defects. The surface of the TRP2 sample degraded, becoming “tacky” developing blisters; the surface of the TRP1 sample blistered and the PVA paint discolored.

The remaining sealants (Teflon tape, heat shrinkable tube and Micro-Isolat) were evaluated in a second phase. For all three materials no saturation of the gypsum samples by resin was observed. The composite components were cut along the length and removed from the core. Figure 4 shows a comparison between a saturated sample without any sealing and a non-saturated sample sealed with Micro-Isolat.

The non-saturated sample still has the normal grayish color of normal gypsum. The saturated sample exhibited a change of color which results from the resin system used.

### 3.2 Influence of additives on the hardening process of gypsum

The chemical bonding of water into the crystalline structure of calcium sulfate is an exothermic reaction. The resulting heat can be measured by a calorimeter (Thermometric TAM Air, Ursinus). The change of magnitude of the maximum heat and the time of occurrence can then be evaluated.

The samples were produced with the modeling plaster. One reference sample of pure gypsum (\( r = 0.5 \)) was compared to a sample (\( r = 0.3 \)) with a lower water/powder ratio. Another sample is prepared with 10% by mass expandable graphite whereas the percentage refers to the plaster powder mass.

All components were prepared in an air tight sample container with a dry mass of 2 g. The expandable graphite was mixed with the plaster powder. For the provision of water an applicator with blender was used (Figure 5). All the testing components were tempered at 20°C in the calorimeter for 1 hour. The water was added to the sample container and mixed for 1 min. The measurement of the exothermic heat continues for 2 hours until completion of the hydration process.

Figure 6 depicts the evolution of the heat of three samples. All three curves show the typical trend of the hardening process of gypsum. A rapid increase of the released heat to the first local maximum occurred within the first two minutes. This is caused by the released wetting heat [6]. After a short period of decrease, which reflects the nucleus formation, the exothermic growth of the crystals and thus the change from hemihydrate to dihydrate took place. This was represented by the rise of the released heat to the maximum. Thereafter the gypsum cooled to room temperature.

Comparing the two graphs of the raw gypsum in Figure 6 it can be seen that the nucleation took longer with less water added and thus the growth started later. The setting of the gypsum was slowed by a smaller amount of water. The hydration of the gyp-
sum proceeded faster and more violently at a water/powder ratio of 0.5 than at the lower water volume of r = 0.3.

The hydration of the gypsum was slightly delayed by the addition of expandable graphite and the heat release was lower than without the additive. This behavior may have been caused by a small release of the acid in the expandable graphite on contact with water. Plank [7] describes that the setting rate of gypsum is delayed by some acids. An acid release of expandable graphite is tested and cannot be detected with a pH-paper. However, it is possible that the amount of acid liberated was below the detection limit of the pH paper used.

Another reason for the delay of setting could be the increased number of solids at a constant amount of water. A certain amount of water is consumed for the wetting of the graphite particles and is thus no longer available for the hydration of the gypsum.

### 3.3 Principle test of the material composition

In a feasibility test the processability was proven. A mixture of the material with a w/p-ratio of 0.5 and 10% by mass expandable graphite was prepared. The mixture was cast into a cylindrical mold. On the resulting mandrel a sealing layer with a heat-shrinkable tube and subsequently 2 layers of braided textile were applied. The preform was saturated by means of a VARI process. After curing the component below the threshold temperature of the expandable graphite the temperature was raised above the threshold temperature. As a result of the expansion, the core was disintegrated and could be removed from the CFRP component. Figure 7 depicts the composite component with internal tooling inside and the composite part itself after removing the mandrel.

### 3.4 Surface quality

The surface quality was defined as a critical requirement for the core system. The optimum achievable surface quality of gypsum and of the mixture (gypsum and expandable graphite) was investigated with a confocal microscope μ-surf. The surface quality of the manufactured CFRP components with different core sealants was evaluated with a Mahr Perthometer M2 according to DIN EN ISO 4288 using the profile method. The measurement methods were chosen due to handling and accuracy reasons.

The values investigated were the mean surface roughness, \(R_a\), and the mean surface height, \(R_z\). The requirement is to achieve a \(R_z\) value of less than 5\(\mu\)m. The presented values are an averaged value of at least three different measurement traces.

#### 3.4.1 Maximum achievable gypsum sample surface quality

Samples with a water powder ratio \(r = 0.5\) and an expandable graphite ratio of 0%, 10% and 20% were prepared on a glass plate. The glass itself was not investigated as with the measuring method of the confocal microscope it was not possible and with the perthometer the glass would sustain damage. For the gypsum sample without additive the mean surface roughness is 0.39\(\mu\)m and for the maximum measured value, the sample with 1% expandable graphite, is 0.72\(\mu\)m. Why the sample with the lowest expandable graphite ratio shows the highest \(R_z\) is not finally clear. The results of \(R_a\) and \(R_z\) are summarized in Table 2.

#### 3.4.2 Achievable CFRP component surface quality

In Section 3.4.1 the maximum achievable surface quality of the component was investigated if the component surface roughness was the same as the core surface roughness. Application of a sealing agent changes the surface quality and the roughness of the CFRP component will be diminished.

The resulting surface roughness values of the CFRP components are depicted in Table 3. The heat-shrinkable tube achieves with \(R_a = 1.96\mu\)m the best value for the mean surface roughness. For the mean surface height the Teflon tape achieves the best value (9.78\(\mu\)m). However, due to the overlapping of the Teflon tape, jumps in thickness occur. Such variance in the roughness height limits the applicability of the Teflon tape significantly. The values are below the value set in the requirements and thus the grades of sealant are sufficient for the application although the use of heat shrinkable tube and Teflon tape is restricted to simpler geometries without convex sections.

#### 3.5 Properties based on the expansion

The combination of gypsum and expandable graphite results in a mixture which is disintegrated due to expansion. The volume change of the disintegrated material is currently unknown. Thus an estimation of the expansion was carried out. In material investigations the raw expandable graphite expands up to 20 times its initial volume.

Gypsum samples (r = 0.5) with different expandable graphite ratios (5%, 10%, 20%) were prepared. The
resulting volume is the bulk volume of the disintegrated material sample.

These samples were tempered in a convection oven to 180°C for two hours. After this time all samples were completely disintegrated. The resulting volume of the pieces of the 5%-sample after disintegration was approximately the same as the initial volume of the sample. The resulting volume of all particles of the sample with 10% is double and the volume of the sample with 20% is three times as large as the initial volume. This would indicate that a clear path for the disintegrated material must be available to exit the CFRP component.

Another observation during these trials was that the particle size decreases with increasing expandable graphite ratios. This result enables the adjustment of the removal opening in the CFRP part.

4 Conclusion and Outlook

A novel removable gypsum-based core material has been developed and tested. The removal from the component was performed successfully. Due to the porous structure of the core material different sealants were investigated. Three of the sealants fulfill the set requirements (resin impermeability, temperature resistance, surface quality). An assessment regarding the influence of the sealants on the geometrical tolerance for core and CFRP part is underway.

As the core material is a multi-material approach the influence of the expandable graphite was investigated regarding the hardening process of the gypsum, with no major influence being detected. Using the profile method the surface quality was classified. The resulting quality exceeded the set requirements.

In further investigations the mechanical properties of the modified gypsum are of particular interest due to loads acting during the preforming and injection processes. The implementation into the process chain and faster removal of the core due to better energy transmission will also be studied further.

![Figure 1. Principle of a rotational hollow component with (b) and without (a) undercut.](image1)

![Figure 2. Principle of processing and removal of the internal tooling.](image2)

![Figure 3. Averaged expansion of the expandable graphite against temperature.](image3)
Figure 4. Comparison of saturated (left) with non-saturated (right) gypsum sample.

Figure 5. Applicator and sample containers of the calorimeter.

Figure 6. Heat evolution as function of time for different water/powder ratios and added expandable graphite.

Figure 7. The cured composite part with (left) and removed (right) internal tooling.

Table 1. Summary of the different phases of calcium sulfate [5].

<table>
<thead>
<tr>
<th>Formula</th>
<th>CaSO₄ · 2H₂O</th>
<th>CaSO₄ · 0.5H₂O</th>
<th>CaSO₄ III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Denotation</td>
<td>Calcium sulfate dihydrate (DH)</td>
<td>Calcium sulfate hemihydrate (HH)</td>
<td>Calcium sulfate anhydrate (AH III)</td>
</tr>
<tr>
<td>Type</td>
<td>-</td>
<td>α-HH</td>
<td>β-AH III</td>
</tr>
<tr>
<td>Chemically bonded water [W-%]</td>
<td></td>
<td>β-HH</td>
<td>β-AH III</td>
</tr>
<tr>
<td>Density [g/cm³]</td>
<td>2.31</td>
<td>α-HH : 2.757</td>
<td>2.580</td>
</tr>
<tr>
<td></td>
<td></td>
<td>β-HH : 2.619</td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Mean surface roughness Rₐ and mean surface height Rₛ of gypsum samples (r=0.5) with different expandable graphite ratios (0%, 1%, 10%, 20%).

<table>
<thead>
<tr>
<th></th>
<th>Rₐ [µm]</th>
<th>Rₛ [µm]</th>
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</thead>
<tbody>
<tr>
<td>Gypsum</td>
<td>0.39</td>
<td>2.23</td>
</tr>
<tr>
<td>Gypsum mixed with expandable graphite (1%)</td>
<td>0.72</td>
<td>3.74</td>
</tr>
<tr>
<td>Gypsum mixed with expandable graphite (10%)</td>
<td>0.57</td>
<td>2.90</td>
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</table>
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<table>
<thead>
<tr>
<th>Gypsum mixed with expandable graphite (20%)</th>
<th>0.54</th>
<th>2.77</th>
</tr>
</thead>
</table>

Table 3. Resulting CFRP mean surface roughness $R_a$ and mean surface height $R_z$ of samples manufactured with different sealants (heat shrinkable tube, Micro-Isolat, Teflon tape).

<table>
<thead>
<tr>
<th></th>
<th>$R_a$ [µm]</th>
<th>$R_z$ [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat shrinkable tube</td>
<td>1.96</td>
<td>11.23</td>
</tr>
<tr>
<td>Micro-Isolat</td>
<td>2.57</td>
<td>13.24</td>
</tr>
<tr>
<td>Teflon tape</td>
<td>1.85</td>
<td>9.78</td>
</tr>
</tbody>
</table>

Acknowledgments

The authors wish to acknowledge financial funding of this project by GE Global Research, Germany. The authors acknowledge the support by the Faculty Graduate Center of Mechanical Engineering of TUM Graduate School at Technical University of Munich, Germany.

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Weinheim: Wiley-VCH Verlag GmbH & Co. KGaA 2005