Fabrication and Mechanical Properties of Silica/Perfluoropolymer Nanocomposites by Direct Melt-Compounding without Surface Modification on Nano-Silica

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Abstract: The authors’ research group has previously developed a novel method for the fabrication of silica/perfluoropolymer nanocomposites, wherein nano-sized silica particles without surface modification were dispersed uniformly through breakdown of loosely packed agglomerates of silica nanoparticles with low fracture strength in a polymer melt during direct melt-compounding. The method consists of two stages: the first stage involves preparation of the loose silica agglomerate, and the second stage involves melt-compounding of a completely hydrophobic perfluoropolymer, PFA (poly(tetrafluoroethylene-co-perfluoropropylvinylether)), with the loose silica agglomerates. By using this simple method without any lipophilic treatment of silica surfaces, silica nanoparticles with a primary diameter of 190 nm could be dispersed uniformly into the PFA matrix. The main purpose of the present study is to evaluate the tensile properties of silica/PFA nanocomposites fabricated by the above method. In order to elucidate the effects of the size of the dispersed silica in the PFA matrix on the properties of the composites, silica/PFA composite samples exhibiting the dispersion of larger-sized silica particle-clusters were fabricated as negative controls exhibiting poor dispersion state of the silica additives. The results obtained under the present experimental conditions showed that the size of the dispersed silica in the PFA matrix exerts a strong influence on the ultimate properties, such as tensile strength and elongation at break, of the composite materials. Furthermore, uniform dispersion of isolated silica nanoparticles was found to improve not only the Young’s modulus but also the ultimate tensile properties of the composite.

Keywords: Nanocomposite, Perfluoropolymer, Silica Nanoparticle, Colloidal Stability, Tensile Property

1. INTRODUCTION

Fluorocarbon polymers are used as tubing, gaskets and valves that are extensively used in various advanced engineering applications, such as chemical plants, medical and semiconductor manufacturing equipments, because of their smoother surfaces, self-lubrication properties and excellent resistance to extremes of temperature and chemical reagents [1-3]. However, since fluorocarbon polymers, in particular, perfluorocopolymers, have much weaker intermolecular interactions than ordinary hydrocarbon polymers [1,2,4,5], their mechanical properties and dimensional stability are becoming critical issues for applications of these polymers. There have been some attempts to improve these characteristics of fluorocarbon polymers by blending the polymers with inorganic fillers [6,7]. Recent developments in medical and advanced engineering fields require the downsizing of fluorocarbon products with corresponding high-reliability. Therefore, a technique that enables uniform dispersion of nanoscale fillers in fluorocarbon polymers must be developed. However, it has generally been considered to be difficult to fabricate fluorocarbon polymer nanocomposites containing inorganic nanofillers for two reasons. The first is the extremely high cohesive force acting between nanosized materials, and the second is the extremely low chemical affinity of the completely hydrophobic fluorocarbon polymer with inorganic fillers that have hydroxyl-rich hydrophilic surfaces. Thus, fluorocarbon polymer composites containing inorganic fillers have been conventionally fabricated only using melt-compounding methods involving additional and complicated chemical treatments such as intercalation of organic swelling agents into clay layers [6] and modification of the silica surface with silane coupling agents [7]. However, thermal decomposition of these chemical agents contained in the composites likely occurs during the melt-compounding and molding processes, since these processes for composites of fluorocarbon polymers, such as PFA (poly(tetrafluoroethylene-co-perfluoropropylvinylether)), with high melting points must be operated at elevated temperature. Moreover, these agents in the composites, in the form of impurities, exert a harmful influence on semiconductor manufacturing clean processes.

Therefore, in order to ensure the purity of fluorocarbon polymer composites to be as high as possible, surface modifiers to achieve good dispersion of the nanofillers should not be used for fabricating the composites.

The authors’ research group has proposed a simple method for dispersing silica nanoparticles into a perfluoropolymer by direct melt-compounding without requiring any surface modification of the silica [8]. For the direct melt-compounding process, in addition to the shear stress induced in the melt-compounded polymer, the critical threshold value necessary for breaking down the...
agglomerates of nanofillers are the main factors that control the dispersion state of the fillers. Therefore, in the authors’ method, loosely packed agglomerates of silica nanoparticles with low fracture strength (loose silica agglomerates) were prepared intentionally by destabilizing an aqueous solution of nano-sized colloidal silica. By melt-compounding PFA with the prepared loose silica agglomerate, silica/PFA nanocomposites exhibiting uniform dispersion of nano-sized silica particles without surface modification could be fabricated through the breakdown of the agglomerates in the PFA polymer melt. However, the characteristics of the fabricated silica/PFA nanocomposites were not discussed in the previous study [8].

The present study was aimed at elucidating the mechanical properties of the silica/PFA composites filled with unmodified silica nanoparticles fabricated by the proposed method. First, using aqueous solutions of nano-sized colloidal silica as starting materials, the silica agglomerates with different strength were prepared from the destabilized colloidal silica dispersion systems via pH control and salt addition. And then, by melt-compounding the PFA with these silica agglomerates, silica/PFA composite samples exhibiting different silica dispersion states were fabricated. The composite samples obtained were subjected to tensile testing. On the basis of the results obtained, the effects of the size and content of the dispersed silica additives in the PFA matrix on the Young’s modulus, tensile strength and elongation at break of the composites are discussed.

2. EXPERIMENTAL DETAILS

2.1. Conditions for control of the silica dispersion state in the PFA matrix

Since the main purpose of this study was to elucidate the relationship between the silica dispersion state in the PFA composite and the mechanical properties of the composite, the authors made attempts to control the silica dispersion state in PFA matrix from a “good” state (i.e. homogeneous distribution of isolated nano-sized silica particles) to a “poor” one (i.e. uneven distribution of unbroken agglomerates as micron-sized silica particle-clusters). The experimental conditions used for the fabrication of the silica/PFA composites exhibiting different dispersion states of silica are summarized in Table 1. In the present study, as shown in this table, three types of nano-silica were used as additives in the PFA-matrix composite. As examples of silica/PFA nanocomposites exhibiting “good” dispersion state of nano-silica (see Fig. 3 (a)), composites A, A-2 and A-3, wherein silica particles with a diameter, $d_{p,\text{Silica}}$ of 190 nm were dispersed uniformly, fabricated with varying silica volume fractions, $V_{silica}$ from 2.8 to 14.4 % in the authors’ previous study [8] were subjected to tensile testing. As negative controls of the silica dispersion state, composites B and C exhibiting “poor” dispersion state of silica (see Figs. 3 (b) and (c)), were fabricated, respectively, using sintered agglomerates of silica particles with $d_{p,\text{Silica}} = 12$ nm with a high fracture strength and commercially available powders of single-nm-sized fused silica. The procedure for the fabrication of composite A, including the preparation of loose silica agglomerate with $d_{p,\text{Silica}} = 190$ nm, was described in the previous paper [8]. In this paper, the procedure for the fabrication of composites B and C will be described in the following sections 2.2 and 2.3.

2.2. Materials for fabrication of silica/PFA composites B and C

The same PFA powders (Teflon® PFA 350J, Du Pont-Mitsui Fluorochemicals Co., Ltd.) used as the matrix for composite A were used as the matrix polymer for composites B and C as well. Commercially available powder of fused silica with $d_{p,\text{Silica}}$ of around 7 nm (AEROSIL® 300, Nippon Aerosil Co., Ltd.) was used as the additive for the fabrication of composite B. For the fabrication of composite C, a commercially available aqueous colloidal solution of spherical SiO$_2$ with $d_{p,\text{Silica}} = 12$ nm (SNOWTEX®-30, Nissan Chemical Industries, Ltd.) was used as the starting material for silica agglomerate preparation.

2.3. Fabrication and characterization of silica/PFA composites B and C

The silica agglomerates used as additives for the fabrication of composite C were prepared from the destabilized colloidal silica dispersion system with $d_{p,\text{Silica}} = 12$ nm under the same conditions of pH, with a value of 4, and KBr concentration (volume ratio of KBr to SiO$_2$ (KBr/SiO$_2$) = 70/30) as that used for the preparation of loose silica agglomerates with $d_{p,\text{Silica}} = 190$ nm [8]. In order to ensure an increase in the agglomerate strength, the silica agglomerates obtained with $d_{p,\text{Silica}} = 12$ nm were sintered at 873 K for 2 h in air. The packing arrangement of the primary silica particles and pore size distribution in the sintered agglomerates were characterized, respectively, by a scanning electron microscope operated at an accelerating voltage of 10-20 kV (SEM; Hitachi S-800, Hitachi, Ltd.) and the volumetric method using nitrogen adsorption (TriStar3000, Micromeritics Instrument Corp.). In addition, a microcompression test (MCT-W500, Shimadzu Co.) was used to evaluate the fracture strength of the agglomerates.

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<table>
<thead>
<tr>
<th>Composite sample No.</th>
<th>Volume fraction, $V_{silica}$/ %</th>
<th>Form in mixing stage with PFA</th>
<th>Diameter of primary particle, $d_{p,\text{Silica}}$/ nm</th>
<th>Starting material</th>
<th>Preparation conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2.8</td>
<td>Non-sintered loose agglomerate</td>
<td>190</td>
<td>Colloidal silica aqueous solution (MP-2040)</td>
<td>pH 4 &amp; (KBr/SiO$_2$) = 70/30</td>
</tr>
<tr>
<td>A-2</td>
<td>7.7</td>
<td>Powder</td>
<td>$\sim$ 7</td>
<td>Colloidal silica aqueous solution (AEROSIL® 300)</td>
<td>pH 4 &amp; (KBr/SiO$_2$) $\sim$ 70/30</td>
</tr>
<tr>
<td>A-3</td>
<td>14.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>2.8</td>
<td>873 K-sintered agglomerate</td>
<td>12</td>
<td>Colloidal silica aqueous solution (SNOWTEX®-30)</td>
<td>pH 4 &amp; (KBr/SiO$_2$) = 70/30</td>
</tr>
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Table 1. Conditions for the fabrication of silica/PFA composites by the direct melt-compounding method.
3. RESULTS AND DISCUSSION

3.1. Pore structure and fracture strength of prepared silica agglomerates

Figure 1 shows SEM micrographs of a selected area on the surface of (a, b) non-sintered and (c) 873 K-sintered agglomerates of silica particles with $d_{p, \text{silica}} = 12$ nm prepared under various conditions. For the non-sintered agglomerates, the structure of the sample prepared from the destabilized colloidal silica dispersion system via pH control and KBr addition (Fig. 1 (a)) was more porous than that of a blank sample of the silica agglomerate prepared from the stable colloidal solution without pH control or KBr addition (Fig. 1 (b)). A similar relationship between the pore structure of the prepared agglomerate and stability of the colloidal silica solution (the starting material) has also been obtained in the case of the non-sintered silica agglomerates with $d_{p, \text{silica}} = 190$ nm [8]. By controlling the stability of the dispersion system in a colloidal silica aqueous solution via pH control and KBr addition, the packing arrangement of the primary silica particles in the agglomerates prepared could be varied from a dense structure to a porous structure. A comparison of Fig. 1 (b) and 1 (c) show that by sintering at 873 K, the primary silica particles forming the agglomerate coalesce and the center-to-center distance of the particles shrinks, resulting in an increase in the interparticle contact area, which is frequently referred to as “sinter neck” formation and growth.

The effects of the porosity of the silica agglomerate with $d_{p, \text{silica}} = 12$ nm on the fracture strength are shown in Fig. 2 together with the results for the agglomerates with $d_{p, \text{silica}} = 190$ nm obtained in the previous study [8]. The value of $\sigma_{f, \text{silica}}$ was determined as the agglomerate tensile strength from the following equation [9]:

$$\sigma_{f, \text{silica}} = \frac{2.8 \cdot F_{R, \text{silica}}}{\pi \cdot d_{p, \text{silica}}}$$

Here, $F_{R, \text{silica}}$ is the load required for the breakdown of the silica agglomerate as measured by the microcompression test, and $d_{p, \text{silica}}$ is the mean diameter of the test pieces of
silica agglomerates, which corresponds to the distance between the points of loading. The porosity of the agglomerates, \( \phi_{Silica} \), was calculated using the results of the pore volume of the silica agglomerates measured by the volumetric method using nitrogen adsorption. In this figure, the theoretical tensile strength of a packed agglomerate of spherical particles derived on the basis of the binding force acting between two particles forming the agglomerate (Rumpf’s equation) [10]:

\[
\sigma_{f,Silica} = \frac{9 \left( 1 - \phi_{Silica} \right)}{8} \frac{A_{H,Silica}}{24 \cdot a \cdot d_{p,Silica}}
\]

was also plotted as a function of \( \phi_{Silica} \) and \( d_{p,Silica} \). In Eq. (2), \( A_{H,Silica} \) is the Hamaker constant of the silica particles and \( a \) is the distance between the surfaces of the two particles in the agglomerate. The value of \( A_{H} \) for fused silica in vacuum or air has been measured and calculated by several research groups [11-13]. There is a slight scatter, but most of the reported values are on the order of the magnitude of \( 10^{-20} \) Joules. Thus, their average, \( 5.5 \times 10^{-20} \) J, was used as an approximate value of \( A_{H,Silica} \) for silica nanoparticles forming the agglomerate in this paper. For the value of \( a \), the adhesion separation distance that is often taken to be around 0.4 nm (single-ångström-ordered constant) for particles in intimate but chemically un-bonded contact with a surface [13-17] was adopted.

The value of \( \sigma_{f,Silica} \) for the sintered silica agglomerate with \( d_{p,Silica} = 12 \) nm was higher than that for the non-sintered one in the case of nearly the same porosity. This is caused by the difference in the agglomerate structure shown in Figs. 1 (b) and (c). The sinter neck formed by strong siloxane (Si-O-Si) bonds during the sintering of the agglomerate leads to an increase in the agglomerate strength. Figure 2 also shows that the agglomerate strength decreases with decreasing diameter of the primary silica particles and increasing porosity which supports the theoretical agglomerate strength as expressed by Eq. (2). However, the experimental results for \( d_{p,Silica} = 12 \) and 190 nm are not in agreement with the theoretical agglomerate strength. All the experimental values of the strength determined from the microcompression test are higher than the corresponding theoretical values. The causes of these deviations of the experimental results from the theoretical strengths were considered, as below. In the previous study [8], the authors concluded that the destabilizing process of the dispersion system in the colloidal silica solution via pH control and KBr addition leads to bridging via potassium ions (K\(^+\)) at a limited number of surface sites on the silica nanoparticles in the destabilized colloidal silica solution, resulting in the formation of three-dimensional networks of silica particles. Deviations of the experimental results from the theoretical strength in Fig. 2 might be caused by the formation of chemical bonds, such as the above-mentioned bridging via K\(^+\), partly between the primary silica particles in the prepared silica agglomerate. Moreover, fracture of the silica agglomerate with non-uniform pore structure (i.e. local existence of some dense packing regions with relatively higher porosity) during the microcompression test is a typical local phenomenon that obeys the weakest link theory, although the porosity of the prepared silica was determined as average data to determine the pore distribution. The difference between the control modes of \( \sigma_{f,Silica} \) and \( \phi_{Silica} \) may also cause a deviation from the theoretical strength of agglomerate derived on the assumption of porous structure with mono-pore size.

In the present study, since fused silica used for the fabrication of composite B is in a powder form, the values of \( \sigma_{f,Silica} \) and \( \phi_{Silica} \) for this sample were not evaluated by the microcompression test and the volumetric method using nitrogen adsorption. It is expected from Fig. 2 that the stress required for the dispersion of the cluster of fused silica powders (AEROSIL® 300) to their primary particles with \( d_{p,Silica} \approx 7 \) nm is a little less than twice as high as the strength of the prepared silica agglomerate with \( d_{p,Silica} = 12 \) nm.

3.2. Dispersion states of silica in fabricated silica/PFA composites

Figure 3 (a) shows a typical example of SEM micrographs of the fracture surfaces of the silica/PFA composite A fabricated by melt-compounding PFA with 2.8 vol.% non-sintered loose silica agglomerates with \( d_{p,Silica} = 190 \) nm described in the previous paper [8]. The silica agglomerate used for the fabrication of composite A has a porosity, \( \phi_{Silica} \), of around 0.45 and the lowest strength among the other silica agglomerates prepared in the present study, as shown in Fig. 2. The micrograph in Fig. 3
(a) clearly shows that the isolated primary silica particles with \( d_{p,Silica} = 190 \text{ nm} \) are dispersed uniformly in the PFA matrix. This implies that the prepared loose silica agglomerates was sufficiently weak for them to be broken down to the primary nanoparticles in the PFA melt by the shear stress induced under the melt-compounding conditions of the previous study [8]. The dispersion states of silica particles in composites A-2 and A-3 have already been described in the previous paper [8] for varying volume fraction of the non-sintered loose silica agglomerates with \( d_{p,Silica} = 190 \text{ nm} \) mixed with PFA. It was found that both composites A-2 with \( V_{f,Silica} = 7.7 \% \) and A-3 with \( V_{f,Silica} = 14.4 \% \), as well as composite A with \( V_{f,Silica} = 2.8 \% \), exhibit a homogeneous distribution of isolated primary silica particles in the PFA matrix.

Figures 3 (b) and (c), respectively, show the dispersion states of silica in the negative control samples of composites B and C fabricated by melt-compounding under the same conditions as used for the fabrication of composite A. These types of silica additives, such as sintered agglomerates and single-nm-sized powders, used for the fabrication of composites B and C are so dense that the additives could not be broken down and they remained as micron-sized large particle-clusters in the melt-compounded PFA. For composites B and C exhibiting poor dispersion of silica, the sizes of the silica particle-clusters remaining in the PFA matrix were examined as follows. The values of the average and the standard deviation of the ESD of the silica particle-clusters in composite B were, respectively, calculated to be 2.0 \( \mu \text{m} \) and 0.9 \( \mu \text{m} \) by analyzing the silica phase appearing as bright regions in the SEM micrographs of the composite surfaces (ex. Fig. 3 (b)). The same analysis of the SEM micrographs of the surfaces of composite C (ex. Fig. 3 (c)) was used to determine the average and the standard deviation to be 41.1 \( \mu \text{m} \) and 18.8 \( \mu \text{m} \), respectively. The results obtained from the above analysis showed that the dispersion state of silica additives in composite C became poorer than that in composite B. This implies that the fracture strength of the sintered agglomerate of silica particles with \( d_{p,Silica} = 12 \text{ nm} \) might be higher than that of the cluster of fused silica powders with \( d_{p,Silica} \approx 7 \text{ nm} \).

On the basis of the results obtained in the authors’ previous [8] and current studies, it is clarified that the dispersion state of silica nanoparticles in the PFA matrix by the direct melt-compounding method is significantly affected by the fracture strength of the particle agglomerates added into the mixer.

### 3.3. Tensile properties of silica/PFA composites

Typical stress (\( \sigma \))-strain (\( \varepsilon \)) curves of the fabricated silica/PFA composites A, B, and C containing the same amounts of silica \( (V_{f,Silica} = 2.8 \%) \) are shown in Fig. 4. The values of (a) the Young’s modulus, \( E_c \), (b) the ultimate tensile strength, \( \sigma_{u,c} \), and (c) the elongation at break, \( \varepsilon_{b,c} \), for these three composites exhibiting different dispersion states of silica additives are shown in Fig. 5 as a function of the mean size of the dispersed silica additives in each composite, \( d_{Silica in PFA} \). In the present study, the value of \( d_{Silica in PFA} \) in the case of composite A was regarded as \( d_{Silica} (= 190 \text{ nm}) \), since this composite clearly exhibited homogeneous distribution of isolated primary silica particles, as shown in Fig. 3 (a). In the case of composites B and C, \( d_{Silica in PFA} \) was evaluated using the average ESD of the silica additive in each composite. In both Figs. 4 and 5, the results for pure PFA prepared by melt-compounding the virgin PFA powders without silica addition were also plotted to compare with the results of the silica/PFA composites.

The stress-strain curves in Fig. 4 display yielding behavior at \( \varepsilon_c \) in the vicinity of 10 % and plastic deformation within the range of \( \varepsilon_c \) from each yielding point to its corresponding breaking point. From Figs. 4 and 5, the Young’s modulus of all composites A, B and C consis-

![Fig. 4. Typical stress-strain curves of pure PFA and silica/PFA composites A, B and C with \( V_{f,Silica} = 2.8 \% \) fabricated under various conditions. The plus (+) symbol shown at the end of each curve represents the breaking point of the test piece.](image)

![Fig. 5. Effects of the size of the dispersed silica additives in the silica/PFA composite with \( V_{f,Silica} = 2.8 \% \) on its (a) Young’s modulus, (b) ultimate tensile strength and (c) elongation at break at room temperature. The tensile properties of composites A, B and C are represented by the closed, half-closed and open circles, respectively. The properties of pure PFA are represented by the hatched regions.](image)
tently show a value higher than that of pure PFA, independent of $d_{\text{Silica}}$ in PFA. These improvements in the modulus are caused by the inclusion of hard silica. On the other hand, increases in $\sigma_{\text{uc}}$ and $\epsilon_{\text{uc}}$ of the silica/PFA composites fabricated in the authors’ previous study [8] with decreasing size of dispersed silica additives in the PFA matrix are observed, in contrast to that observed for $E_c$. In particular, composite A exhibiting a homogeneous distribution of isolated primary silica particles with $d_{p, \text{Silica}} = 190$ nm in the PFA matrix yielded a larger elongation at break than in the case of pure PFA.

The ultimate tensile properties, such as elongation at break, of polymer materials have a tendency to decrease, by the inclusion of micron-sized metal oxide additives although the elastic modulus increases [18-20]. However, composite A fabricated by the authors’ research group [8], wherein isolated silica nanoparticles are dispersed uniformly, was found to possess both a larger Young’s modulus as well as a larger elongation at break than pure PFA.

The values of $E_c$ and $\epsilon_{\text{uc}}$ for the silica/PFA nanocomposite A are plotted as a function of the silica volume fraction in Fig. 6 (a) and (b), respectively. This figure shows the results obtained from the stress-strain curves of composites A, A-2 and A-3 exhibiting similar good nano-dispersion of silica. The Young’s modulus increased with increasing $V_{\text{Silica}}$. The value of $\epsilon_{\text{uc}}$ was found to initially increase with increasing $V_{\text{Silica}}$ and showed a value of around 400% at $V_{\text{Silica}} = 2.8$ %. Although further addition of silica decreased the value of $\epsilon_{\text{uc}}$, this value could be kept at a high level close to the elongation at break of pure PFA even in the case of 14.4 vol.% of silica in the polymeric matrix, wherein isolated silica nanoparticles are dispersed uniformly, was found to possess both a larger Young’s modulus as well as a larger elongation at break than pure PFA.

The mechanical properties of silica/PFA nanocomposites on the (a) Young’s modulus and (b) elongation at break at room temperature. The tensile properties of composites A, A-2 and A-3 exhibiting homogeneous distribution of isolated primary silica particles with $d_{p, \text{Silica}} = 190$ nm in the PFA matrix are represented by closed circles. The properties of pure PFA are represented by the hatched regions.

4. CONCLUSIONS

The mechanical properties of silica/PFA nanocompo-
sites were investigated by a simple melt-compounding method without any complicated lipophilic treatments of the silica surfaces. The following results were obtained under the present experimental conditions:

(1) With regard to the fabrication method for the silica/PFA nanocomposites, the fracture strength of an agglomerate of silica nanoparticles was found to determine the dispersion state of silica in the PFA matrix. This indicated that a reduction in the agglomerate strength of silica before the melt-compounding stage is one of the key strategies for obtaining uniform dispersion of the silica nanoparticles in this method.

(2) The size of the dispersed silica in the PFA matrix exerts a strong influence on the ultimate tensile properties, such as tensile strength and elongation at break, of the composite materials.

(3) In the case of dispersion of micron-sized silica in the PFA matrix, the ultimate tensile properties of the composites were lower than those of the pure PFA although the Young’s modulus became higher than that of the pure PFA.

(4) In the case of uniform dispersion of nano-sized silica in the PFA matrix, in addition to the modulus, the ultimate properties were successfully improved compared to the pure PFA. This implies that the sites of the isolated silica nanoparticles distributed homogeneously in the PFA matrix did not act as points of stress concentration during various external loadings.

(5) The method proposed by the authors’ research group was found to be a simple fabrication process for high performance silica/PFA nanocomposites exhibiting excellent mechanical properties.

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