On-chip fabrication of micrometer-size super-hemispherical and spherical optical devices from molten glass droplets

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Surface-tension molding (StM) and localized-laser heating (LLH) techniques were developed for on-chip fabrication of micrometer-size super-hemispherical glass (μ-SSG), which has a truncated spherical shape, and spherical glass. The optical functionalities of these glasses as solid immersion lenses (SILs) and whispering gallery mode (WGM) optical resonators were demonstrated. In the StM method, glass particles on a glassy carbon substrate were heat-treated under an H₂/N₂ atmosphere and the glass particles were melted and deformed into super-hemispherical shapes that were determined by the wetting property of glass melt on the substrate. The contact angle of the droplet ranged from 120 to 167° depending on the glass composition. We found that the shape of a μ-SSG with a composition of 20Na₂O−10CaO−70SiO₂ (in mol %) satisfied the optical condition of a SIL and demonstrated super-resolution for μ-SSG fabricated by StM. The LLH technique enables us to make a nearly perfect sphere on a substrate. A micrometer-size Nd³⁺-doped tellurite glass particle on a transparent substrate was irradiated by a continuous-wave (CW) laser with a wavelength near 810 nm and a power of more than 150 mW, and only the particle was heated and melted into a spherical shape on a transparent substrate at room temperature. The obtained spherical glass had a very smooth spherical surface with high sphericity. The Nd³⁺-doped microsphere showed WGM resonances to free-space direct pumping of the CW laser with a wavelength of 790–820 nm, and laser emissions were observed with a threshold of a few-milliwatts. Effects of add-on structures, e.g., a terrace or a bubble, on the laser characteristics of the microsphere were also investigated. Both structures acted as an entrance for pumping light and resulted in laser thresholds lower than 1 mW. The terrace structure reduced the emission modes and showed quasi-single mode laser emission. The bubble-containing microsphere laser realized broad excitation spectra due to the modified WGM. These results indicate that a spherical glass with a smooth surface formed from a molten glass droplet on a chip has excellent potential in the field of optics and photonics.

Key-words: Glass melt, Surface tension, Laser heating, Solid immersion lens, Near-field optics, Spherical laser, Whispering gallery mode

1. Introduction

Inorganic glass materials have been widely used for optic and photonic applications not only due to their excellent optical properties, such as a wide optical window, various refractive indexes, and dispersion, but also due to their high mechanical/chemical/thermal durability and excellent formability. This glass is often made using the melt-quenching method. Glass melt above the glass transition temperature (Tg) is formed into the desired shape and quenched to obtain optical components such as fibers, lenses, or plates. This material can possess an incredibly smooth surface, regarded as the liquid free surface, which is ideal for low-loss optical devices if a non-contact melt-quenching process is used. A liquid droplet tends to form spherical shapes automatically due to surface tension. This spherical surface is fundamental for optical components such as lenses and mirrors, in which the light is converged or dispersed by refraction or reflection at the spherical plane. Moreover, if an ideal spherical shape with a free surface is obtained, the sphere can exhibit interesting optical phenomena such as super-resolution and optical resonance due to photon confinement.¹³⁻³

Glass spheres can easily be formed by quenching a floating molten glass droplet. However, in this method, precise control of their physical properties, such as the size, shape, and position of the spheres, is impossible. An on-chip fabrication process is desirable to control the physical properties and is preferred for subsequent align-
ment for incorporation into optical systems. In this review, we focus on a super-hemispherical, which is a truncated sphere, and a true sphere with a very smooth surface, which can act as a super-resolution lens and a high-quality optical resonator respectively. Two different on-chip fabrication processes of micrometer-size glass having a spherical surface have been developed, and their optical functionalities have been demonstrated. One is a surface-tension molding (STM) method preparing micrometer-size super-hemispherical glass (μ-SIL) using the wetting property of a glass melt on a substrate. The other is a localized-laser heating (LLH) technique fabricating a true sphere on a transparent substrate using the dewetting phenomenon due to local heating. The optical functionalities of these glasses as solid immersion lenses (SILs) and whispering gallery mode (WGM) optical resonators are demonstrated.

1.1 Solid immersion lens

In 1990, Kino et al. developed an SIL for microscopy with a super-resolution circumventing the optical diffraction limit. Figure 1 shows a schematic illustration of a super-hemispherical SIL (super-SIL) for which aberration-free focus occurs. This focusing occurs when the sphere is truncated to a thickness of \(a(1 + 1/n)\), where \(a\) is the radius of the curvature and \(n\) is the refractive index of the super-SIL. A focal point is located on the flat bottom surface when the incident light is focused on a point below a distance of \(n \times a\) from the center of the sphere. Because the wavelength is reduced by a factor of \(n\) and the effective numerical aperture is increased by \(n\), the spatial resolution of a super-SIL is increased by a factor of \(n^2\) at most. Many types of applications can benefit from SILs, such as microscopes, optical data storage, lithography, Raman imaging, and photoluminescence imaging.

The probe shape must be controlled precisely to realize super-resolution. For a super-SIL, the aberrations and allowances of the dimensions are as follows:

\[
|b| < \lambda / 4 \sqrt{n^2 - 1}, \tag{1}
\]

\[
|d| < \lambda / 4n(n\sqrt{n^2 - 1} - n^2 + 1), \tag{2}
\]

where \(b\) is the aspheric error, \(d\) is the thickness error, and \(\lambda\) is the wavelength of the light in air. When \(\lambda = 405\) nm and \(n = 1.5\), \(|b| < 91\) nm and \(|d| < 158\) nm are required to achieve the theoretical spatial resolution. It is difficult to fabricate the ideal shape of a super-SIL; however, the shape allowance of a hemispherical SIL is much larger and can be produced via a semiconductor fabrication process. In the STM method, the aspheric error is reduced when the particle size is sufficiently small to ignore the deformation due to gravity and the thickness can be readily controlled by the wetting property between the glass melt and the substrate via the glass composition.

1.2 WGM optical resonator

A small dielectric sphere can encapsulate photons due to WGMs, as shown in Figure 2. WGM optical resonators have received considerable attention for many years due to their potential to be used in many applications such as low-threshold microlasers, high-precision frequency combs, and high-sensitivity chemical and biological sensors. These benefits are derived from their high quality factor and small mode volume. According to the Lorenz–Mie theory, the parameter \(Q_{\text{sc}}\) of a microsphere with a radius \(a\) is given by

\[
Q_{\text{sc}} = \frac{2}{\lambda^2} \sum_{n=0}^{\infty} (2n + 1) |(a_n|^2 + |b_n|^2), \tag{3}
\]

\[
a_n = j_{mn}(x) [j_{mx}(j_{nx}x) - m^2 j_{mx}(j_{nx}x) \phi_{nx}(x)], \tag{4}
\]

\[
b_n = j_{mn}(x) [j_{mx}(j_{nx}x) - m^2 j_{mx}(j_{nx}x) \phi_{nx}(x)], \tag{5}
\]

where \(x = 2\pi a / \lambda\) is the size parameter, \(\lambda\) is the wavelength of the light in the surrounding medium, and \(m\) is the relative refractive index, which is the ratio between the refractive index of the sphere, \(n\), and that of the surrounding medium.

![Figure 1. Schematic illustration of a super-SIL.](image)

![Figure 2. Schematic illustration of the WGM resonator.](image)
2. StM technique

2.1 Molten glass droplet on a solid substrate

The shape of a liquid droplet on the surface of a flat solid is determined by the Young–Dupré equation:

\[
\cos \theta = \frac{\gamma_S - \gamma_{SL}}{\gamma_L},
\]

where \(\gamma_S\), \(\gamma_L\), and \(\gamma_{SL}\) are the surface tensions of the solid and the liquid and the interfacial tension between the solid and the liquid, respectively. \(\theta\) is the contact angle. A partially truncated sphere (\(\theta > 90^\circ\)) of a molten glass droplet can be obtained when an appropriate material, such as a carbon material, is chosen as a solid substrate. A perfectly spherical droplet cannot be achieved on a substrate under gravity because the spherical part is slightly distorted by gravity and forms a flat surface with \(\theta\), which is determined by Eq. (6). For a small droplet, gravity can be ignored because the surface tension of the liquid is much larger than the gravitational force. In the case of a silicate glass melt, the capillary length \(k^{-1} = (\gamma_S/\rho g)^{1/2}\) is approximately 3 mm, where \(\rho\) and \(g\) are the density and the gravitational acceleration, respectively, and \(\gamma_S\) has a typical value of 0.3 N/m for a silicate glass melt. Therefore, when the size of a molten glass droplet is reduced to a micrometer size, an almost perfect spherical shape can be formed. The contour of a droplet can be calculated using the Young–Laplace equation. The calculated oblateness, 1 – \(a_t/a_e\), ranges from approximately 0.001 to 0.003 for few-micron to few-hundred-micron diameter spheres. Here \(a_t\) and \(a_e\) represent the radii of the curvature at the top and at the equatorial plane, respectively. Therefore, we can fabricate a nearly perfect spherical shape with a micrometer-size molten glass droplet on a substrate having a low wettability for glass melt.

2.2 Fabrication of super-hemispherical glass using the StM technique

Figure 3 shows a schematic illustration of the StM process. In our study, glassy carbon was chosen as the substrate material for the StM treatments because its wettability for oxide-glass melts is suitable to obtain a droplet with a contact angle larger than 90° and because its thermal durability under a reducing atmosphere is sufficient to prevent chemical reactions with oxide glasses. The substrate was polished to optical grade, and glass particles with sizes of 20–45 \(\mu\)m on the substrate were heat-treated at a temperature above the glass softening point under an \(H_2/N_2\) atmosphere. As shown in the scanning electron microscope (SEM) image in Fig. 3, the shape of the glass changed into a truncated sphere.

Figure 4 shows the relationship between the refractive index and the contact angle of the samples obtained via StM treatments, and the insets show SEM images of the samples. Figure 4 includes results of soda lime borosilicate glass and optical glasses; Barium flint glass (BaF10) and \(La_2O-ZnO-B_2O_3-SiO_2\) glass (LZBS1 and LZBS2). The glass particles had a super-hemispherical shape with an incredibly smooth surface, which is suitable for optical use. The contact angles ranged from 120° to 167° depending on the glass composition. These results indicate that the shape of the glass can be controlled from nearly hemispherical to spherical by choosing the glass composition.

The size and position of the \(\mu\)-SSGs can also be controlled using the optical contact between the thin glass foil and the glassy carbon, photolithography, buffered hydrofluoric acid, and subsequent Ar+ etching and the StM technique. Figure 5 shows an SEM image of a fabricated \(\mu\)-SSG array. Each \(\mu\)-SSG had the same contact angle \(\theta\) because \(\theta\) is determined by the unique nature of the glass melt and the substrate material, that is, the balance of the surface/internal tensions.
2.3 SIL functionalities of super-hemispherical glass fabricated via StM

In Fig. 4, the solid line indicates the SIL optical condition:

\[ n = \frac{a}{h - a} = \frac{1}{\cos \theta}, \]  

(7)

where \( n \), \( a \), \( h \) and \( \theta \) are refractive index, radius of curvature in spherical part, height and contact angle of the SIL, respectively. The green area in Fig. 4 is the adaptive area considered from the margin of the flat bottom surface of the SIL calculated from Eq. (2). This figure indicates that we fabricated super-SILs at glass compositions of approximately \( x = 0 \) and 47 in the series of 20Na2O–10CaO–xB2O3–(70 – x)SiO2 (in mol %).

We demonstrated the SIL functionality of the \( \mu \)-SSGs of the 20Na2O–10CaO–70SiO2 (x = 0) glass fabricated via StM.\(^{13}\) The \( \mu \)-SSG was put on an observation sample, which is an integrated circuit (IC) chip having a fine structure whose atomic force microscope image and cross-sectional profile are shown in Figs. 6(a) and 6(b), respectively. Figure 6(c) shows an optical microphotograph of the \( \mu \)-SSG on the IC chip. In Fig. 6(e), the focus of the objective lens of the microscope is located on the surface of the IC chip. Figure 6(d) is an optical photograph of the surface of the IC chip observed through the \( \mu \)-SSG. In Fig. 6(d), grooves with a depth of approximately 20 nm in the center of the 1.3-\( \mu \)m period stripes were seen through the \( \mu \)-SSG. This photo was obtained due to the evanescent wave from the flat bottom surface of the \( \mu \)-SSG. Therefore, we confirmed the SIL functionality of the \( \mu \)-SSG fabricated via the StM technique.

In the StM process, the morphology and composition of the flat bottom part in the SIL can be modified by changing the surface condition of the substrate. The addition of a thin gold film to the SIL can realize both high-contrast and high-resolution imaging\(^{35}\) because gold nanostructures are highly sensitive to refractive index changes in the surrounding medium due to surface plasmons. We fabricated the SIL with gold nanoparticles (AuNPs) via the StM technique using a thin gold film coated glassy carbon substrate.\(^{16}\) Figures 7(a) and 7(b) show a schematic illustration of the fabrication process and SEM images of the SIL with AuNPs and (b) SEM images of the fabricated sample.

![Fig. 5. SEM image of a super-spherical glass array fabricated via a combination of optical contact, photolithography, and StM.](image1)

![Fig. 6. (a) AFM image of an IC chip, (b) cross-sectional profile of the white line in panel (a), (c) optical photograph of a super-spherical glass on an IC chip, and (d) optical photograph of an IC chip through a super-spherical glass.](image2)

![Fig. 7. (a) Schematic illustration of the fabrication process for glass SIL with AuNPs and (b) SEM images of the fabricated sample.](image3)
These results indicate that the SIL with AuNPs can be a powerful tool for both high-sensitivity and high-resolution optical microscopy.

2.4 WGM resonance and laser oscillation from μ-SSGs

The μ-SSGs have a spherical part, which could act as a WGM resonator. We can confirm the WGM resonance from the μ-SSG by doping the emitting center with rare-earth ions in the glass matrix and measuring their fluorescent spectra.

Figure 9 shows the fluorescence spectra from 1 mol % Eu³⁺ doped 20Na₂O–10CaO–70SiO₂ (in mol %) mother glass and μ-SSGs fabricated using the StM method under irradiation with a continuous-wave (CW) Ar⁺ laser at a wavelength of 514.5 nm. The excitation laser was introduced parallel to the equatorial plane of the spherical part, and the focal point was placed near the edge of the equatorial line using a 50× objective lens. In the spectra of the μ-SSGs with 9.7 and 6.2-μm diameters, periodic sharp peaks were observed on the broader fluorescence spectrum of the ⁵D₀→⁷F₂,1,0 transition of Eu³⁺. The comb-shaped lines above the spectra from the μ-SSGs indicate the grouping of the spike signals by mode number. The mode spacings from the μ-SSGs with diameters of 9.7 and 6.2 μm were 245 and 380 cm⁻¹, respectively. These values agree well with the theoretical mode spacing \(\Delta\nu\) given by

\[
\Delta\nu = \frac{1}{2\pi a} \tan^{-1}\left(\frac{m^2 - 1}{1}\right)^{1/2}.
\]

The constant mode spacing indicates that the μ-SSGs function as optical microcavities to encapsulate the optical waves inside their spherical parts. The \(Q\) value estimated from the full width at half maxima was 10⁴, which was limited by the spectral resolution of the monochromator. These results indicate that the StM technique enables us to fabricate WGM resonators on a substrate whose wetting property of the glass melt is sufficiently low.

3. LLH technique

When a molten glass droplet is placed on a flat substrate, the contact interface between the droplet and the substrate forms a flat glass surface after cooling below \(T_{g}\). In the StM process, pinning of the contact line sometimes distorts the spherical part and reduces the \(Q\) factors of the resonances. For a high-\(Q\) WGM cavity, a perfectness of the circular equatorial portion is significant. However, nonadhesion melting of a glass droplet on a substrate under gravity is severe; therefore, the on-chip formation of a perfect sphere of glass appears to be impossible. However, the LLH technique can realize nonadhesion melting of a glass droplet due to the dewetting phenomenon induced by the temperature gradient. When a liquid droplet falls onto a molten pool or solid substrate and a temperature difference exists between them, coalescence or wetting of the droplet is prevented. The reason for the prevention of coalescence and wetting is attributed to Marangoni floating, which induces a thin air film under the droplet.

In the LLH technique, the same dewetting phenomenon may occur and a nonadhesive glass droplet on the substrate can be realized. Typically, a transition-metal or rare-earth-ion-doped glass particle on a transparent substrate such as silica glass is irradiated by a high-power CW laser and only the glass particle is heated and melted; then, a nearly perfect glass sphere can be fabricated on the substrate.

3.1 Fabrication of micrometer-size glass spheres via LLH

Figure 10 shows a typical optical system for the LLH process. A high-power CW laser is focused onto a glass
particle containing an absorption center on a substrate that is transparent at the laser wavelength. Table 1 summarizes the properties of the glass microspheres fabricated via the LLH technique. All the glasses contain rare-earth ions as heat sources derived from the absorption of laser light and subsequent non-radiative transitions. The substrate materials do not have optical absorption at the laser wavelength and are not heated by laser irradiation. Not only low softening temperature glasses such as tellurite or bismuth borate but also high-\(T_s\) glasses such as silicate can be formed into spherical shapes via the LLH technique.

Figure 11 shows side-view optical snapshots of a glass particle with a composition of 2.0Nd\(^{3+}\)-doped 10K\(_2\)O–10WO\(_3\)–80TeO\(_2\) (in mol %) during the LLH process using a high-speed camera. A CW-Ti:Sapphire laser with a wavelength of 806 nm and a power of 268 mW irradiates the glass particles on the silica glass substrate through an objective lens (100x, NA0.80). When the laser starts to irradiate, a glass particle is formed into a spherical shape without a flat part within 0.06 s. First, the molten glass droplet contains many bubbles, which convect inside the droplet. The bubbles can be moved and gathered by tuning the position and power density of the laser spot. These bubbles can be used for the entrance of a pumping light and to modify the WGM resonance, as shown in Section 3.3. Figure 12 shows a side-view SEM image of the fabricated glass microsphere. The microsphere has a good spherical shape with an oblateness \((1 - a_1/a_e)\) larger than 0.99 and a very smooth surface. We confirmed that microspheres with sizes ranging from 4 \(\mu\)m to 0.8 mm can be obtained via the LLH technique. The ellipticity, which was estimated from the top-view optical-microscope photographs, of the equatorial (horizontal) plane in the sphere was less than 1\%. These results indicate that the LLH technique has a high potential to realize on-chip fabrication of microspheres for integrated optical devices.

### 3.2 Laser oscillation of Nd\(^{3+}\)-doped tellurite glass microspheres

The laser characteristics of the Nd\(^{3+}\)-doped K\(_2\)O–WO\(_3\)–TeO\(_2\) glass microspheres fabricated via the LLH technique were investigated. The laser wavelengths were 806 and 980 nm for Nd\(^{3+}\) and Yb\(^{3+}\), respectively. \(n_{Hg:Ne}\) and \(n_d\) are the refractive index at the wavelength of 633 and 587.6 nm, respectively. \(n_{calc}\) is estimated by using INTERGLAD\(^{2(2)}\)

| Glass composition (in mol %) | Dopants for laser absorption | Glass transition temperature | Refractive index |
|-----------------------------|-----------------------------|----------------------------|-----------------|
| 10K\(_2\)O–10WO\(_3\)–80TeO\(_2\) | Nd\(^{3+}\) or Yb\(^{3+}\) | 310°C | \(n_{Hg:Ne}:2.05\) |
| 23K\(_2\)O–15Nb\(_2\)O\(_5\)–62TeO\(_2\) | Yb\(^{3+}\) | 347°C | \(n_{calc}:1.9\) |
| 30Bi\(_2\)O\(_3\)–30ZnO–40B\(_2\)O\(_3\) | Nd\(^{3+}\) | 419°C | \(n_{calc}:1.97\) |
| 20Na\(_2\)O–10CaO–70SiO\(_2\) | Nd\(^{3+}\) | 515°C | \(n_{calc}:1.515\) |
nique were examined using a direct pumping setup with a tunable CW-Ti:Sapphire laser.\textsuperscript{21) Figure 13(a)} shows an example of a lasing spectra from a microsphere with a diameter of 31\,\mu m pumped with the approximately 800\,nm band of the $^4I_{9/2} \rightarrow ^4F_{5/2}$ transition of Nd$^{3+}$. The pumping position was the edge of the microsphere, and the pumping power was much less than that of the LLH process. The lasing peak due to the $^4F_{3/2} \rightarrow ^4I_{11/2}$ transition was observed from the sphere. The intensity of the signal, marked by a closed circle in Fig. 13(a), is plotted against the pumping power in Fig. 13(b). A sudden increase in the peak intensity was observed at 5.69 mW, which is the laser threshold of the sphere; such nonlinear behavior indicates laser oscillation of the glass microsphere.

3.3 Improvements in the laser characteristics of glass microspheres via add-on structures

How to realize the coupling between a high-$Q$ WGM resonator and pumping/emission light is a significant problem for practical use. Tapered-fiber coupling is one of the best techniques to achieve a high coupling efficiency of more than 99\%. However, optical device with fine dimension and its precise position control are necessary for efficient coupling; a few-micrometer-diameter optical fiber in air approaches to microcavity within a few hundred nanometer. The direct pumping technique is one of the most straightforward coupling techniques; however, its efficiency is quite low because the radiation loss of a spherical microcavity is isotropic and low.\textsuperscript{43) An add-on structure can modify WGMs and improve the coupling efficiency without critically degrading the $Q$ factor.

We developed two types of add-on structures for the spherical resonator: a terrace and an air bubble. SEM images of microspheres with an added terrace and air bubble are shown in Figs. 14(a) and 14(b), respectively. The terrace structure, which is made of an organic–inorganic hybrid material, was fabricated via the capillary method.\textsuperscript{21),44)–46) The air bubble was introduced by controlling the laser irradiation conditions during the LLH process.\textsuperscript{20),22) Figure 15 shows the emission spectra from the microsphere before and after terrace formation. The peak position of the no-terrace microsphere is in good agreement with the theoretical $Q_{\text{ sca}}$ calculated from Eq. (3) based on Mie theory. Conversely, the emission peak positions of the terrace microsphere are not the same as the theoretical ones. The number of peaks from the terrace microsphere was lower than that from the as-prepared sample, and nearly single mode emission was realized. The reason for the quasi-single mode laser emission may be that the terrace structure modifies the WGM by partially decreasing the relative refractive index. The laser thresholds of the as-prepared and terrace microspheres were 1.3 mW pumped at the vicinity of the sphere and 0.6 mW pumped at the terrace. The laser threshold of the terrace microsphere was lower than that of the uncoated microsphere. These results indicate that the terrace structure enables not only modification of the WGMs but also a decrease in the laser
threshold due to an increase in the coupling efficiency between the cavity and the free-space beam.

Figure 16 shows the excitation and emission spectra from the bubble-containing microsphere. The diameters of the microsphere and the bubble were 20 and 1.6 μm, respectively. When the vicinity of the sphere was pumped, the excitation spectra of the vertical and horizontal polarizations coincided well with the peaks of the theoretical TM and TE modes, respectively. In the case of pumping at the bubble, many peaks were observed in both polarization directions. However, in the emission spectra, all the peak positions agreed with the theoretical ones; therefore, the emission modes of the bubble-containing microsphere are same as a non-add-on microsphere having a high Q factor. These results indicate that the introduction of the bubble to the microspherical resonator modifies the WGM modes and enables coupling with light at any pumping wavelength and polarization direction in the absorption band of the emission center. Therefore, broad pumping light sources such as lamps, LEDs, and solar light can be used for high-Q optical resonators.

4. Conclusions

We developed two types of on-chip fabrication techniques for micrometer-size spherical glass for optical devices.

In the StM technique, molten glass droplets on a glassy carbon substrate form truncated microspheres and their contact angles are controllable using the wetting property via the glass composition. The truncated microspheres, which are fabricated from 20Na2O–10CaO–70SiO2 glass, function as evanescent field optics to visualize fine structures with 20-nm depths. A combination of AuNPs and the SIL was also fabricated and realized both high-sensitivity and high-resolution optical imaging. Moreover, WGM resonances from the truncated microspheres were demonstrated.

The LLH technique enables the fabrication of a nearly perfect sphere on a substrate transparent to the laser light. This microsphere exhibits low-threshold laser action based on high-Q WGMs. The addition of a terrace structure reduces the laser threshold and modifies the emission modes. The addition of an air-bubble structure lowers the laser threshold and broadens the excitation spectra due to non-WGM excitation.

The StM and LLH techniques are based on the characteristics of glass materials, which are solidified from a supercooled liquid without crystallization, and enable the formation of spherical glass that can achieve high optical functionalities such as super resolution and high-Q resonators. Spherical glasses with incredibly smooth surfaces are promising devices that will be novel tools in the field of optics and photonics.

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