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Microstructure and mechanical properties of Zn25Al3Cu based composites with large Al\textsubscript{2}O\textsubscript{3} particles at room and elevated temperatures

Microstructures and compressive properties of Zn25Al3Cu alloy and Zn25Al3Cu/Al\textsubscript{2}O\textsubscript{3} particulate composites with large reinforcing particles (250 \textmu m) were examined. The composites were obtained by the compocasting technique through infiltration of 3, 8 and 16 wt.\% Al\textsubscript{2}O\textsubscript{3} particles into the semi-solid metal matrix. The influence of temperature in the range from 70 to 170 °C on compressive yield strength of the matrix alloy (as-cast and thixocast Zn25Al3Cu alloy) and the composites was investigated. Above 70 °C compressive yield strength of all materials decreases, but the rate of decrease is different for the matrix alloy (as-cast and thixo-cast) and composites. It was found that the abrupt decrease in compressive yield strength of the matrix alloy occurred at temperatures higher than 70 °C, whereas composites retained relatively high values of compressive yield strength till the end of the testing temperature range regardless of the amount of reinforcing particles.

Keywords: Metal-matrix composites; Zinc–aluminum alloys; Al\textsubscript{2}O\textsubscript{3} particles; Compocasting; Compressive yield strength

1. Introduction

Among the zinc-based foundry alloys the alloys with relatively high content of aluminum (zinc–aluminum or ZA alloys) are distinguished by excellent bearing properties. The alloys are designated 8, 12 and 27 because of their approximated aluminum content. These alloys are also characterized by favorable combinations of physical, mechanical and technological properties (low melting point, high strength, fine machining and good tribological properties, excellent castability) as well as by low manufacturing costs. The alloys are suitable for a wide application range (industrial findings and hardware, pressure-tight housings, sleeve bearings, thrust washers and wear plates). However, ZA alloys are only applicable at operating conditions up to 80 °C, due to a deterioration of mechanical properties at elevated temperatures [1].

ZA27 alloy is the lightest of the ZA alloys and offers excellent bearing and wear resistance properties. The alloy is characterized by a dendritic microstructure [2, 3] and a non-uniform distribution of chemical elements in the alloy phases [4]. ZA27 alloy solidifies in a wide temperature range and is suitable for processing in the semi-solid state. It was shown that a non-dendritic structure as well as favorable mechanical properties of the alloy could be achieved by thixocasting [5–8] with subsequent heat treatment [9, 10]. The rheological behavior of the semi-solid processing of this alloy was studied by Lehuy et al. [11].

Compocasting [12] has been used to produce ZA-based composites. This technique is characterized by infiltration of reinforcing particles into the semi-solid metal matrix. It was shown that physical and a number of mechanical characteristics (elasticity modulus, hardness, wear resistance, linear thermal expansion coefficient) of ZA27 alloy-based composites with addition of Al\textsubscript{2}O\textsubscript{3}, SiC [13–15] and ZrO\textsubscript{2} [16] particles were better with respect to the matrix alloy at room temperature. Cornie et al. [13] carried out an investigation of mechanical properties of ZA27 alloy strengthened by SiC particles and Al\textsubscript{2}O\textsubscript{3} whiskers at room temperature. However, the results at elevated temperatures have not been reported.

Compressive properties of commercial bearing alloys at room and elevated temperatures (about 100 °C) are of exceptional importance for their application in the manufacturing of sliding bearings [17]. ZA27 alloy is a suitable bearing material for heavy and light load pressures with low and high surface speeds. To overcome the deterioration of the alloy mechanical properties at elevated temperatures particulate composites with base ZA27 alloy were developed.

It is well-known that the size of reinforcing particles significantly affects mechanical properties of composites. At uniform particle distribution, better mechanical properties were achieved by using small particles [18, 19]. Hard particles such as SiC, Al\textsubscript{2}O\textsubscript{3} and ZrO\textsubscript{2} [13, 14, 20] and soft particles such as graphite and coke dust [21, 22] were used for producing composites with base ZA27 alloy. The size of particles was mostly from 50 to 150 \textmu m. There are only a few papers [15, 23–25] concerning composites with reinforcing particles larger than 200 \textmu m. Addition of these particles to the matrix alloy offers several advantages which are as follows:

- infiltration of particles into the semi-solid melt of matrix alloy can be easily performed with a dosator, due to the particles size
mixing of composite semi-solid melt during the compocasting process can be carried out in the laminar flow regime with minimum vortex and small energy consumption,

- large (250 µm) particles are not prone to agglomeration in contrast to small (12 µm) particles [15]. Small particles exhibit significant tendency towards formation of B type agglomerates as was defined by Tseng [26].

- it is possible to develop a compocasting procedure with reduced time of mixing and favorable distribution of reinforcing particles in the metal matrix. Thus, in relation to composites with small reinforcing particles, except that a more uniform distribution of particles may be obtained, the use of large particles in composites production may be more favorable from the economic point of view because of shorter process duration and lower energy consumption.

Besides, it was recently shown that the wear resistance of the AZ27/Al2O3 particulate composites with large particles (250 µm) was higher with respect to as-cast AZ27 alloy at room temperature [23, 24].

In this work large Al2O3 particles were used for processing of composites with Zn25Al3Cu matrix alloy. Compressive properties of the matrix alloy and the composites were studied. The effect of the reinforcing particles amount (3, 8 and 16 wt.% Al2O3) on the compressive yield strength of the composites was examined at room and elevated temperatures (from 70 to 170 °C). Microstructural development of the composite matrix during thixocasting and compocasting process was also studied considering the influence of matrix morphology on the mechanical properties of composites.

The chemical composition of Zn25Al3Cu alloy is very similar to that of AZ27 alloy except that copper content is slightly higher than recommended by EN 12844 [27]. Murphy’s investigations related to AZ27 alloy [3] and the results reported in [28] for Zn25Al3Cu alloy indicate microstructural similarity and close mechanical properties of both alloys. Therefore, experimental results obtained within this work can be compared with the results for AZ27 alloy based composites with ceramic particles.

2. Experimental procedure

2.1. Test samples

Zn25Al3Cu alloy was processed in the Department of Materials Science “Vinca” Institute. The nominal composition (in wt.%) of the alloy was as follows: 27 Al, 3 Cu and Zn remainder. The alloy casting was performed at 580 °C into a steel mold preheated up to 100 °C. The as-cast samples were cylindrical, 36 mm in diameter and 120 mm in height.

2.1.1. Thixocasting

As-cast Zn25Al3Cu alloy was charged into the crucible of an electro-resistance furnace [29]. The alloy was melted and preheated up to 550 °C to clean the slag from the melt surface. The melt was left to cool down to 485 °C at 5 K min⁻¹ cooling rate (approximately isothermal regime) and the active part of a paddle stirrer was then immersed into the melt. Mixing of the matrix semi-solid melt was performed within the temperature range between liquidus and solidus temperatures with a gradual increase in stirrer rotation speed. A stationary mixing regime (i.e. mixing at constant temperature and constant mixing rate) was achieved at a rotation speed of 450 rpm and at 461 °C. The duration of mixing of the semi-solid melt during thixocasting was 10 min. After that the melt was cooled down to 450 °C and then poured into the steel mold preheated to 300 °C. Obtained thixocast castings were cylindrical, 36 mm in diameter and 120 mm in height. Hot-pressing at 230 °C and 100 MPa was applied after cooling in order to reduce porosity of the thixocast castings.

2.1.2. Compocasting

Thixocasting represents the first stage of the compocasting process. Large Al2O3 particles (3, 8 and 16 wt.%) were infiltrated continuously with mixing into the semi-solid melt of the matrix alloy. No previous chemical preparation of the Al2O3 particles was performed. Al2O3 particles were preheated at 200 °C for 1 h in order to eliminate moisture and reduce thermal shock during infiltration. Reinforcing particles were added into the zone next to the shaft of the active part of the stirrer. Compared to 450 rpm performed during thixocasting, mixing was done at relatively lower speeds, i.e. at 200 rpm for 3 and 8 wt.% Al2O3, and 100 rpm when 16 wt.% Al2O3 particles was added. Lower speeds were applied in order to prevent segregation of reinforcing particles in the peripheral zones of castings as well as to avoid air penetration into the melt. Temperatures of particle mixing and infiltration time were dependent on the reinforcing particle content as presented in Table 1.

The composite semi-solid melt was poured into the steel mold preheated to 300 °C. The composite samples were cylindrical with 36 mm in diameter and 120 mm in height. Hot-pressing of all composites was done at 230 °C and 250 MPa. Note that there is a difference in pressure applied during hot-pressing of thixocast and composite samples. Namely, previous experimental results [30] showed that porosity of composites was completely eliminated when applying 250 MPa at 230 °C during hot-pressing.

<table>
<thead>
<tr>
<th>( \delta (\mu m) )</th>
<th>250</th>
</tr>
</thead>
<tbody>
<tr>
<td>( G (%) )</td>
<td>3</td>
</tr>
<tr>
<td>( T (°C) )</td>
<td>461</td>
</tr>
<tr>
<td>( t_{in} (min) )</td>
<td>4</td>
</tr>
<tr>
<td>( t_{out} (min) )</td>
<td>30</td>
</tr>
</tbody>
</table>

The notation in Table 1 is as follows: \( \delta \) – average size of Al2O3 particles, \( G \) – weight fraction of Al2O3 particles, \( T \) – temperature of mixing, \( t_{in} \) - infiltration time and \( t_{out} \) – total time of mixing.
2.2. Characterization of the matrix alloy and the composites

2.2.1. Microstructural investigations

Optical microscopy (OM) and scanning electronic microscopy (SEM) were used as well as energy dispersive spectroscopy (EDS) for microstructural characterization of the matrix alloy and composites.

Samples were machine-cut from ingots and then ground and polished. Grinding was performed with 80, 360 and 600 grits emery paper, while polishing was done using polishing cloth and polishing paste with Al₂O₃ particles. Polished samples were used for SEM and EDS analysis, whereas samples for OM examination were polished or etched in 9% v/v HNO₃.

2.2.2. Compression tests and hardness

Compression tests were performed at room (20°C) and elevated temperatures: 70, 120 and 170°C. This temperature interval is similar to that applied for tensile tests of zinc-based alloys [1], since there are no data in the literature on the temperature range for compression tests. The operating temperature was maintained within ±1°C. The tests were done on cylindrical specimens (4 mm in diameter and 8 mm in height) using a tensile test machine with a hot chamber. The specimens were heated for 20 min prior to compression testing. A loading rate of 1 mm min⁻¹ was applied. Three specimens of the as-cast as well as of thixocast matrix alloy and three specimens of each composite type were tested and the average values of compressive yield strength was calculated.

The room temperature hardness tests were conducted in accordance with ASTM E10 [31] using a Brinnell hardness tester with a ball indenter of 2.5 mm diameter and a load of 62.5 kg. The load was applied for 30 s. Tests were performed on specimens 36 mm in diameter and 10 mm in height. Five hardness readings were taken for each specimen at different locations.

3. Results and discussion

3.1. Microstructure

3.1.1. Zn25Al3Cu as-cast

The general appearance of the matrix alloy as-cast microstructure can be seen in Fig. 1a and b. Alloys which solidify through peritectic reaction during cooling are characterized by this kind of structure. A typical dendritic structure indicates non-equilibrium solidification. The dendrite core consists of α phase, while α + η phase mixture prevails at the dendrite periphery. The interdendritic space is filled with η phase. The presence of η phase (CuZn₄) was also noticed. This phase is distributed in the form of mainly eutectic particles within η phase (Fig. 1b). It has been reported that η phase had a beneficial effect on the mechanical and wear properties of Zn25Al3Cu alloy, particularly after certain heat treatment regimes [3]. Prolonged etching caused considerable dissolution of η phase resulting in a vague appearance of other constituents of Zn25Al3Cu alloy (Fig. 1b).

Varying concentrations of zinc and copper increase reach the maximum and then decrease while the variation of aluminum concentration is the opposite. It is noticeable that copper concentration is significantly lower in regard to zinc concentration. These results show that that dendrite cores are rich in aluminum while the amount of zinc is rather high in the interdendritic space.

Fig. 1. Microstructure of as-cast Zn25Al3Cu. (a) SEM, polished. DC – dendrite core; DP – dendrite periphery; IDS – interdendritic space; (b) OM detail of η phase, etched; (c) variation of chemical composition along the L line (Fig. 1a). EDS: 1 – Al; 2 – Zn; 3 – Cu.
3.1.2. Zn25Al3Cu thixocast

The microstructure of thixocast Zn25Al3Cu alloy is shown in Fig. 2a–d. Zn25Al3Cu alloy was used as a metal matrix in the compocasting process. During this process the dendritic structure of the alloy was transformed into a non-dendritic one. Large primary particles nearly elliptic in shape consist of core (C in Fig. 2b) and periphery (P in Fig. 2b). The periphery consists of an $\alpha + \eta$ phase mixture. The primary particles are surrounded by an interspace phase (IS in Fig. 2b) that is filled with $\eta$ phase. Additionally, small islands of $\alpha + \eta$ phase mixture are visible inside the cores of primary particles (Fig. 2b and Fig. 2c-detail). The elliptic form of the primary particles and their size indicate that the microstructural transformation was completed in accordance with the scheme proposed by Flemings [5].

Variations of chemical composition along the L line (Fig. 2d) are shown in Fig. 2e. It can be seen that the distribution of elements in the thixocast microstructure is very similar to their distribution in the as-cast microstructure of the matrix alloy (see Fig. 1c).

![Microstructure of thixocast Zn25Al3Cu, SEM, polished.](image)

**Fig. 2.** Microstructure of thixocast Zn25Al3Cu, SEM, polished. (a) transformed structure; (b) microconstituents: C – core; P – periphery; IS – interspace; (c) a detail: islands of $\alpha + \eta$ phase mixture within $\alpha$ phase; (d) line position for EDS; (e) EDS. 1 – Al; 2 – Zn; 3 – Cu.
Table 2. Size of $\alpha$ phase particles ferret and volume fraction of phases in Zn25Al3Cu alloy.

<table>
<thead>
<tr>
<th>Zn25Al3Cu</th>
<th>The largest ferret* of $\alpha$ phase (µm) (Average value/range)</th>
<th>Volume fraction (vol.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>20 (8–39)</td>
<td>$\alpha$</td>
</tr>
<tr>
<td>Thixocast</td>
<td>87(28–120)</td>
<td>$\alpha + \eta$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\eta$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>11.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>74.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>56.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>13.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15.4</td>
</tr>
</tbody>
</table>

* Connected with the largest diameter of the particle.

In spite of radical change of morphology during thixo-casting process it is obvious that a change in chemical composition within individual microconstituents did not occur. The regrouping of existing phases in the microstructure took place and this may be described as a morphological change. The microstructure of the thixocast Zn25Al3Cu alloy is similar to the microstructure of other alloys subjected to thixocasting: Sn-15Pb [6], Al-Cu, Al–Si and Al–Ni [7]. The size of $\alpha$ phase particles increased during thixo-casting process as did the volume fraction of $\alpha$ phase compared to the as-cast microstructure. This is demonstrated in Table 2. It can be also noticed that volume fraction of $\alpha + \eta$ phase mixture significantly decreased, while the change in $\eta$ phase volume fraction was rather small.

Agglomeration of the primary particles of $\alpha$ phase occurred during thixo-casting. The agglomerates retained elliptic shape due to the influence of shear forces created by mixing [5]. Their de-agglomeration was prevented because of an instantaneous increase in viscosity after the mixing of the semi-solid melt was terminated and with the onset of cooling. With further cooling a peritectic reaction took place between these elliptic agglomerates of $\alpha$ phase and the remaining melt creating $\beta$ phase. $\beta$ phase was transformed by eutectoid reaction into the $\alpha + \eta$ phase mixture.

3.1.3. Zn25Al3Cu/Al2O3 composites

The microstructure of composites is shown in Fig. 3a–c. Large Al2O3 particles are uniformly distributed in the matrix alloy regardless of their overall content. Segregation of the reinforcing particles was not noticed and neither was their tendency to form agglomerates.

Details of the microstructure of the composite with 3 wt.% Al2O3 particles are shown in Fig. 4a and b. The microcracks along the matrix/particle interface (A) and the microcracks starting from the reinforcing particle towards the matrix (B) can be seen more clearly at higher magnification (Fig. 4a). The microcracks are also observed at the boundary between the $\eta$ phase and $\alpha + \eta$ phase mixture (Fig. 4b).

The microstructure of an etched sample of the composite with 3 wt.% Al2O3 particles is shown in Fig. 5a. Large primary particles of $\alpha$ phase are visible in the microstructure of the composite matrix that is very similar to the microstructure of the thixoalloy matrix. Porosity at the interface between a large reinforcing particle (RP) and the matrix was not observed at this level of applied magnification.

A detail of microstructure corresponding to the composite with 16 wt.% Al2O3 is presented in Fig. 5b. A fractured Al2O3 particle is shown broken into smaller fragments kept together by the metal matrix. No scattered fragments detached into the matrix were observed. Fragments of parti-
3.2. Mechanical properties

3.2.1. Compressive yield strength and hardness at room temperature

Experimental results of compressive yield strength in dependence on temperature are presented in Fig. 6. The compressive yield strength of 420 MPa for as-cast Zn25Al3Cu alloy (Fig. 6, curve 1) is somewhat higher (8%) than the value prescribed by the standard for ZA27 alloy [27]. This increase is probably due to a slight increase in the copper content in the investigated alloy compared to the standard ZA27 alloy [27] since the addition of copper favors the formation of \( \alpha + \eta \) phase. The compressive yield strength of thixo-cast Zn25Al3Cu alloy (Fig. 6, curve 2) is lower compared to the as-cast alloy. A coarse-grained structure was formed during thixo-casting and the volume fraction of \( \alpha + \eta \) phase (see Table 2) was significantly decreased resulting in a decrease of strength of the thixo-cast alloy.

On the other side, compressive yield strengths of Zn25Al3Cu/Al2O3 composites (Fig. 6, curves 3–5) are lower with respect to the as-cast alloy and similar to that of the thixo-cast alloy. Values of compressive yield strength of the composites are lower regardless of the amount of reinforcing particles although one might expect higher values for the composites bearing in mind the mechanism of composite hardening proposed by Arsenault [25]. According to this mechanism, density of dislocations increases with rein-

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Fig. 4. Microstructure of Zn25Al3Cu/Al2O3 composite (3 wt.% Al2O3), SEM, polished. (a) interface and microcracks; (b) microcracks development.

Fig. 5. Microstructure of Zn25Al3Cu/Al2O3 composites, OM, etched. (a) 3 wt.% Al2O3, RP – reinforcing particle; (b) 16 wt.% Al2O3, a detail – fractured Al2O3 particle.

Fig. 6. Compressive yield strength dependence on temperature. 1 – Zn25Al3Cu as-cast; 2 – Zn25Al3Cu thixo-cast; 3 – Zn25Al3Cu + 3 wt.% Al2O3; 4 – Zn25Al3Cu + 8 wt.% Al2O3; 5 – Zn25Al3Cu + 16 wt.% Al2O3.
forcing particle content and decreases with strengthening particle size, but is always higher compared to the non-strengthened matrix alloy. Within this work the lower compressive strength of composites may be ascribed to the structure of the composite matrix being quite similar to the structure of thixocast alloy. In addition, attention should be paid to the negative effect of microcracks formed either along the matrix/particle interface or microcracks situated at the boundaries between $\alpha + \eta$ phase mixture and $\eta$ phase (see Fig. 4a and b, respectively). This effect has also previously been reported [33].

The results of Brinell hardness testing are presented in Table 3. These results confirmed that the hardness of the composites increased with the increase in reinforcing particle content. The hardness of the composites with 3 and 8 wt.% $\text{Al}_2\text{O}_3$ particles is similar to the hardness of the matrix alloy (as-cast and thixocast), while hardness of the composite with 16 wt.% $\text{Al}_2\text{O}_3$ particles is higher. This is in accordance with results reported by Cornie et al. [13].

Table 3. Hardness of Zn25Al3Cu alloy and Zn25Al3Cu/Al2O3 particulate composites at room temperature.

<table>
<thead>
<tr>
<th>Material</th>
<th>Hardness (BHN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn25Al3Cu, as-cast</td>
<td>111</td>
</tr>
<tr>
<td>Zn25Al3Cu, thixocast</td>
<td>94</td>
</tr>
<tr>
<td>Zn25Al3Cu + 3 wt.% $\text{Al}_2\text{O}_3$</td>
<td>85</td>
</tr>
<tr>
<td>Zn25Al3Cu + 8 wt.% $\text{Al}_2\text{O}_3$</td>
<td>113</td>
</tr>
<tr>
<td>Zn25Al3Cu + 16 wt.% $\text{Al}_2\text{O}_3$</td>
<td>118</td>
</tr>
</tbody>
</table>

4. Conclusions

On the basis of the results presented, the following conclusions are proposed:

1. Large $\text{Al}_2\text{O}_3$ particles are uniformly distributed in the matrix Zn25Al3Cu alloy regardless of the content of reinforcing particles. Porosity of the particle/matrix interface and segregation of the reinforcing particles were not detected at the level of applied magnification.

2. The cracks in the large $\text{Al}_2\text{O}_3$ particles could be induced by thermal stress. As there is a large mismatch between thermal expansion coefficients of the matrix alloy and these reinforcing particles, a significant thermal stress will exist during cooling of the Zn25Al3Cu/$\text{Al}_2\text{O}_3$ composites. The large $\text{Al}_2\text{O}_3$ particles which have low thermal expansion coefficient will sustain a tensile stress, and may crack during cooling.

3. Hardness of Zn25Al3Cu/Al2O3 particulate composites increases with increasing in reinforcing particles content.

4. Zn25Al3Cu alloy both as-cast and thixocast exhibits higher compressive yield strength with respect to Zn25Al3Cu/Al2O3 composites at room temperature. In the temperature range from 70 to 170°C compressive yield strength of the matrix alloy and the composites decreases but the rate of decrease is different. An abrupt decrease in compressive yield strength of the matrix alloy occurred at temperatures higher than 70°C, whereas composites with large $\text{Al}_2\text{O}_3$ particles retained relatively high values of compressive yield strength which is similar for all the composites in the whole range of testing temperatures (from 70°C to 170°C). Compressive yield strength of the composite with 16 wt.% $\text{Al}_2\text{O}_3$ at 170°C is somewhat higher compared to composites with lower $\text{Al}_2\text{O}_3$ particle content.

Deterioration of compressive yield strength of the matrix alloy above 70°C originates from the effect of thermally activated processes which are coupled with an external load. Climbing is the basic mechanism of dislocation motion under the influence of an external load and elevated temperatures [34]. This statement is valid for pure metals when $T > 0.5 T_m$ (where $T$ is the testing temperature and $T_m$ is the melting temperature in Kelvin), but as a first approximation may be applied in the case of ZA27 alloy. The melting temperature of ZA27 alloy is 373°C ($T_m = 646$ K) [1] and if the testing temperature is 100°C ($T = 373 K$), which roughly equals to the middle part of the test temperature range within this study, then $T = 0.57 T_m$. At temperatures above 70°C easier movement of dislocations in the matrix alloy is enabled by diffusion processes resulting in a rapid decrease in compressive yield strength.

Although the size of $\text{Al}_2\text{O}_3$ particles used in this study was rather high (250 μm) the effect of these particles on the matrix strengthening cannot be neglected. The density of dislocations which might arise as a result of different thermal expansion of the metal matrix and $\text{Al}_2\text{O}_3$ particles is, nevertheless, higher in composites than in the matrix alloy. Increasing content of reinforcing particles induces a higher density of dislocations. Being hard these $\text{Al}_2\text{O}_3$ particles act as barriers to the movement of dislocations within the metal matrix aggravating their mobility. The net effect is a slow decrease in compressive strength of the composites at elevated temperatures. For the composite with 16 wt.% $\text{Al}_2\text{O}_3$ particles these effects are more pronounced at 170°C. A similar conclusion was reached by Seah et al. [21] studying ZA27 alloy strengthened with rather large SiC particles (up to 150 μm).

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