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Application optimized compression induced solidification

In this investigation, a possibility for shortening the cycle time in the manufacture of thick-walled optical components by compression induced solidification (CIS) was studied in a numerical manner and experimentally tested for validity. The test specimens produced with a reduced cycle time were compared with the manufacturing processes commonly used for such components, i.e. injection molding, injection-compression molding and conventional CIS, with regard to dimensional accuracy, internal stresses and microstructural impression. It could be shown that by delaying the heating of the mold, the adapting time and thus the cycle time of the process can be reduced by up to 55 %, while maintaining the same high quality of the components.

Anwendungsoptimierte Druckverfestigung

In dieser Untersuchung wurde eine Möglichkeit zur Verkürzung der Zykluszeit bei der Herstellung dickwandiger optischer Bauteile durch Druckverfestigung simulativ betrachtet und experimentell auf ihre Gültigkeit geprüft. Die mit verkürzter Zykluszeit hergestellten Probekörper wurden mit den für derartige Bauteile gängigen Herstellverfahren Spritzgießen, Spritzprägen sowie konventionelle Druckverfestigung hinsichtlich Maßhaltigkeit, inneren Spannungen und Mikrostrukturabformung verglichen. Es konnte gezeigt werden, dass durch ein verzögertes Heizen des Werkzeugs die Angleichzeit und damit die Zykluszeit des Prozesses um bis zu 55 % reduziert werden kann, bei gleichbleibend hoher Qualität der Bauteile.

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1 INTRODUCTION

In the low-price segment of non-imaging optical lenses, the material glass has been largely replaced by optically amorphous polymers [1]. The reasons for this are the low-cost and highly automated production of large quantities by injection molding (IM), the freedom in design of the components and the possibilities of functional integration at a lower specific weight [2, 3]. However, increasing electrification and digitization are driving the demand for imaging lenses [4] for precise acquisition and processing of the resulting data volumes, as they are often used in sensors or scanning systems [5] in the automotive, aerospace, medical technology and automation sectors [6]. In the field of such highprecision applications, polymer lenses still have significant disadvantages compared to glass lenses in terms of dimensional accuracy [7], molding accuracy of microstructures and negative optical properties, such as birefringence [8] or process-related fluctuations in refractive indices across the components cross-section [9]. This is due to the interaction of the materialinherent properties of the polymer with the manufacturing conditions in the IM process. The comparatively low thermal conductivity of the polymer leads to a two-phase cooling process in the edge and core area after injection of the melt into the mold, in which the edge area has already solidified, whereas the core area is still liquid, Figure 1.



Figure 1: Cooling simulation of an injection molded thick walled optical lens [10]

The shrinkage in the core area can be compensated in the subsequent holding pressure phase until the gate and the surrounding thinner part areas freeze. Since the molten polymer has a shrinkage coefficient about three times higher than the solid, this leads to geometrical imperfections e.g. sink marks and undesirable material imperfections e.g. stress birefringence [11, 12], which greatly reduces the imaging accuracy of the optical components. This difficulty is shown in Figure 2 with a pvt-diagram. Up to now, this problem has been counteracted at the expense of sustainability by oversized runner systems, in which high holding pressures can be applied over a long period of time. In combination with the low thermal conductivity of the polymer, this results in cycle times of up to 20 min [13] for the IM of thick-walled optical lenses, which relativizes the economic efficiency of the process [14]. In addition, the high holding pressures can lead to orientations in the gate area of the molded part, which reduces the optical quality of the lens [12, 15].



Temperature T

Figure 2: pvT-diagram of the IM cycle of thick walled optical lenses [16]

In order to optimize the inhomogeneous pressure distribution during the holding pressure phase and thus counteract orientations and residual stresses as well as sink marks and poor dimensional accuracy, injection-compression molding (ICM) is often used for thick-walled components [17]. This process is also used in the field of thin-wall IM and microtechnology to ensure the holding pressure effect in the area of very fine structures or thin-walled component areas with a high surface-to-volume ratio [18, 19]. In this process, the melt is usually injected slowly and gently into a partially opened mold, thus reducing molecular orientation in the gate area [19]. Due to a subsequent compression step as a result of the closing movement of the mold, the holding pressure effect is almost hydrostatic and flat over the entire surface of the cavity. The aforementioned effects of stress birefringence as well as dimensional deviations and sink marks can thus be reduced. Since the progressive solidification of the component from the edge areas to the core area increasingly stabilizes the component and thus prevents shrinkage compensation in the core area during the cooling process, the ICM process leads to an improvement of the aforementioned component effects compared to IM, but by no means to their complete elimination [20, 21]. In addition, the problem of the cooling time due to the low thermal conductivity still remains.

The state of the art with regard to reducing the cycle time in the manufacture of thick-walled optical lenses is multilayer IM [16]. For this purpose, a pre-molded part is first injection molded, which features a thinner wall thickness than the later component. Afterwards the pre-molded part is overmolded in several steps in the same mold until the desired final wall thickness is achieved, whereby surface effects and sink marks can be compensated by overmolding. Due to the quadratic dependence of the cooling time on the wall thickness, the potential for reducing the total cycle time when the thick-walled component is produced in layers is over 40% [22]. However, the holding pressure and the melting temperature of the injected components can induce stresses in the boundary layer between the pre-molded part and the overmolded part, which also reduces the optical quality of the components and complicates the process control [16]. In addition, the significantly higher investment costs for the required turntable or index plate molds compared to conventional IM or ICM processes, as well as the higher machine costs, mean that this process only becomes economically viable for high production volumes. Multilayer IM is used, for example, in the production of non-imaging lenses for automotive headlights [16] or LED collimators for lighting optics [23].

An approach to solving the problems in the manufacture of thick-walled components, which is being researched at the Institute of Polymer Technology, uses the pressure dependence of the glass transition in order to decouple the solidification of the polymer from cooling. The transition of an amorphous polymer melt into the glassy solid state is determined, among other things, by the restriction of the mobility of the polymer chains [24]. This is normally achieved by a reduction of the free volume between the polymer chains as the component cools, so that the polymer solidifies within a glass transition

temperature range [25, 26]. Compression induced solidification (CIS) uses the effect of restricting the free volume between the interpenetrating polymer chains and thus their mobility not by cooling but by a sufficiently high pressure at a constant temperature [27]. Thus the polymer can be solidified by applying pressure at temperatures above the glass transition at which it is still flowable under atmospheric conditions [28]. The course of CIS is shown schematically in Figure 3 using a pvT diagram. After the isochoric injection process (1+2), the melt is adapted to the temperature of the mold (3). As soon as there are no more temperature inhomogeneities within the component, solidification takes place by compression of the melt (4). The solidified melt is then cooled under isobaric conditions until it is decompressed at a temperature below the glass transition (5) and cooled to room temperature under ambient pressure (6).



Temperature T

Figure 3: pvT-diagram of the CIS cycle of thick walled optical lenses [16]

In order to avoid the negative effects resulting from shrinkage differences between the edge and core area, it is necessary to solidify the polymer melt simultaneously over the entire cross section of the component. Within the framework of CIS, this is only possible if the component has a homogeneous temperature across the cross-section before the application of pressure [29]. The pressure dependence of the glass transition is material-specific and is 0.3 K/MPa for polycarbonate at a glass transition temperature of approx. 145 °C [28]. This relationship illustrates that too high pressures would be required in relation to mold strength, in order to solidify the polycarbonate - which is usually processed at 280 °C - directly after injection. In order to work with moderate mold pressures, the mold is therefore kept at a temperature of approx. 20 °C above the glass transition by means of dynamic mold temperature control. The melt is cooled to this temperature during an adapting time after injection. The pressure, required for simultaneous solidification, is then applied by a compression stamp. In a subsequent cooling step, the component is cooled down to demolding temperature while maintaining the pressure at a constant level. As a result of the simultaneous solidification of the polymer, the component shrinks within this cooling step over the entire cross-section with the same coefficient of thermal expansion as a solid body, whereby residual stresses, dimensional deviations and sink marks can be avoided [32].

The disadvantage of this process is the time taken for the melt to reach mold temperature. Although the negative effects of the conventional process variants can be reduced, the low thermal conductivity of the polymer melt leads to long cycle times compared with multi-layer IM, due to the previously necessary adapting time. In the context of this investigation, the possibility of a reduction of the adapting time by delayed heating of the dynamically tempered mold cavity within the framework of the CIS cycle is to be investigated on a numerical basis. Subsequently, the cycle time optimized process (CIS_CTO) will be compared experimentally with the existing CIS process as well as with the conventional processes of IM and ICM with regard to dimensional accuracy, stress birefringence and accuracy of molding microstructures.

2 EXPERIMENTAL

2.1 Processing

Due to the high pressure dependence of the glass transition, a polycarbonate type Makrolon LED 2245 (Covestro AG, Leverkusen, Germany) with a glass transition temperature of 145 °C (ISO 11357-1/-2) [30] was selected for the investigation. The test specimens were produced on an IM machine type KM 110 CX SP380 (KraussMaffei Technologies GmbH, Munich, Germany) in a two-cavity injection compression mold (Viaoptic GmbH, Wetzlar, Germany) with dynamic mold temperature control designed for the CIS process.

The test specimen consists of a lens (sphere) with a microstructure and a truncated cone (disc) with a change in wall thickness. The geometry of the test specimen and the measuring positions of the simulated temperature curves are shown in Figure 4.



Figure 4: Specimen dimensions, all dimensions in mm; black dots indicate the measuring positions for the simulation

The processing parameters for the production of the reference components IM, ICM and CIS are given in Table 1. The settings of the CIS cycle result from the pressure dependence of the glass transition of polycarbonate (0.3 K/MPa [28]) and the maximum possible mold temperature of 170 °C. In order to shift the glass transition to a temperature of 185 °C, and thus well above the mold temperature, a pressure of 1333 bar is required. The adapting time was determined by the simulation. The high mold temperature above the glass transition enabled gentle injection at low speed to avoid shear induced orientations without causing imperfections on the part surface. For the reference processes, 3 test specimens were produced for each variation and 5 test specimens were produced for each variation and 5 test specimens were processes IM and ICM was carried out with an enlarged gate channel geometry for a better holding pressure effect and filling process as the mold temperature is colder here.

Parameter	IM	CIM	CIS
Melt temperature [°C]	280	280	280
Mold temperature [°C]	110	110	170/60 °C *
Packing pressure [bar]	1300	1300	1300
Injection velocity [mm/s]	100	100	6
Cooling time [s]	300	300	100
Compression velocity [mm/s]	-	1	0,15
Compression gap [mm]		1,5	1,5
Adapting time [s]			480

* dynamic mold temperature control

Table1: Manufacturing parameters for the reference specimen

The cycle time of the reference parts IM and ICM is made up of the time to open and close the mold (20 s) as well as the injection time and holding pressure/cooling time and resulted in approx. 320 s for both processes. The cycle time of the CIS process also includes the time for heating the mold (165 s) as well as the adapting time, the compression time (10 s) and the time required to reduce the clamping force (20 s) and adds up to approx. 780 s. The production of the test specimens with cycle time reduction was carried out in the same way as for the CIS specimens, with the difference that here the adaptating time was reduced by injecting into the cold mold at 60 °C and then heating the mold according to the simulation results.

2.2 Simulation

The simulation software Moldflow Synergy 2019 (Autodesk, Mill Valley CA, USA) was used to determine the adapting time and to investigate the possibility of cycle time reduction as a result of delayed heating. The material data for the simulation were obtained from the Moldflow database (see Appendix). The meshing as a 3D volume model was carried out with tetrahedra of the global edge length 1 mm with 1.2 million elements. A melt temperature of 280 °C was selected for all simulations. In order to represent the real conditions, the temperature curve during heating and cooling of the mold was measured with thermocouples in the cavity of the mold and selected as input variable for the mold temperature in the simulation. The calculated temperature curves were determined on the specimen disc at the locations of the gate, component top side, component bottom side and component core. In order to determine the

ideal start for heating the mold, the time to start heating after injection was varied between 10 s and 60 s with a time interval of $\Delta t = 10$ s.

2.3 Characterization

The microstructure impression was examined using microscopic images of the Fresnel structure of the convex lens on a scanning electron microscope type Ultra Plus (Carl Zeiss Microscopy GmbH, Jena, Germany). The images were taken on one component at a time with a secondary electron detector at an acceleration voltage of 10 kV under 250-fold magnification.

The evaluation of the residual stresses and orientations was carried out on three samples of the reference components and on five samples of the CIS components by calculating the maximum principal stress difference according to the main equation of photoelasticity [31]:

$$\sigma_1 - \sigma_2 = \frac{S}{d} * n$$

where n is the maximum isochromatic order, S is the photoelasticity constant and d is the component thickness. The maximum isochromatic order was determined by photoelasticity images in a photoelasticity tester (Dr. Heinrich Schneider Messtechnik GmbH. Kreuznach, Bad Germany) under monochromatic light. The effect of birefringence is used here, in which polarized light is split into two partial waves along the main stress axes of the component, which pass through the body at different speeds. This phase difference can be made visible as an interference pattern by a second polarization filter, which is arranged perpendicular to the polarization direction of the light. The interference pattern is composed of the lines with the same phase difference (isochromats) and the lines where the polarization direction of the incident light coincides with the main stress direction (isoclines). Since this phase difference can reach a multiple of the wavelength of polarized light, the isochromats are divided into different orders. This isochromatic order is directly proportional to the degree of residual stress and the molecular orientations within the component. The effect of birefringence is material-specific, which is why the photoelasticity constant is used to calculate the maximum principal stress difference. For polycarbonate this constant is 7.5 - 10 N/mm. [31] A constant of 8 N/mm was chosen for the investigations [33]. In addition, the phase shift is dependent on the distance of light travelled in the component, which is why the specimen disc with a flat surface of constant thickness were used for the examination of the maximum principal stress difference. The thickness was determined by three measurements with a micrometer for each component.

The evaluation of the dimensional accuracy was carried out on three components of the reference components and on five components of the CIS components by measuring the height of the truncated cone of the test specimen disc five times in each case. As the largest sink mark is expected in the centre of the component due to the high material accumulation, a dial gauge holder

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was constructed in the course of the investigation for exact centring of the dial gauge over the centre of the component. A dial gauge (Mitutoyo Corp., Kawasaki, Japan) with a resolution of 0.001 mm was used for the measurement. The designed holder is shown in Figure 5. The measurement is carried out by three-point contact of the holder on the outer ring of the specimen. The holder was zeroed using a ground plate.



Figure 5: Dimensions of the dial gauge holder for determining the dimensional accuracy

The nominal dimension was determined by 5 measurements with the same device directly in the mold at 20 °C, 60 °C and 180 °C mold temperature. The results are shown in Table 2.

Mold temperature [°C]	Nominal dimension [mm]	Standard deviation [mm]
20	4.0248	7.48E-4
60	4.0244	1.62E-3
180	4.0302	7.48E-4

 Table 2:
 Nominal dimensions of the height of the truncated cone in the mold for different mold temperatures

3 RESULTS AND DISCUSSION

3.1 Simulation

Figure 6 shows the calculated temperature curves at the positions core and gate as well as top and bottom side of the component for the conventional CIS cycle. The time for heating the mold to the target temperature is about 165 s. After an initial shear heating of the melt during the injection phase it takes about 480 s until it has cooled down to mold temperature in all investigated part areas. From the start of cooling, it takes a further 100 s until the component has cooled down to demolding temperature of 138 °C. The process design of the CIS cycle for the experiments described in Chapter 2 was based on these results. Taking into account the time required to close the mold and to increase and decrease the clamping force, the cycle time of the CIS cycle is 780 s.



Figure 6: Calculated temperature curves of the measuring positions: gate, component top side, component bottom side, component core and the measured curve of mold temperature for the conventional CIS cycle

The aim of cycle time reduction is to maximize the heat flow from the component to the mold by injecting the melt into an initially cold mold and then successively reducing the heat flow by delayed heating until the component core and mold have the same temperature at the end of the heating cycle. The results for cycle time reduction have been evaluated for the different delay times at the gate and core positions and are shown in Figure 7.



Figure 7: Calculated temperature curves for different heating delay times Left: gate, right: component core

It can be seen that if heating is delayed by more than 20 s, the temperature in the area of the gate undershoots the demolding temperature of 138 °C. In addition, more time is needed to bring the supercooled melt back to mold temperature. The component core, on the other hand, cools down more rapidly the later the start of heating is set. As a compromise the shortest cycle time is thus achieved with 20 s delayed heating. The simulation of the calculated temperatures of this cycle is shown in Figure 8. Taking into account the closing and opening times of the mold, the calculated minimum cycle time is 340 s. This results in a calculated reduction of the cycle time from of approx. 55 % compared to the conventional CIS process (780 s).



Figure 8: Calculated temperature curves of the measuring positions: gate, component top side, component bottom side, component core and the measured curve of mold temperature for the cycle time optimized CIS cycle (delay time: 20 s)

3.2 Experiment

In order to validate this drastic reduction in cycle time, the processes of CIS and cycle-time optimized CIS (CIS_CTO) shown in Figures 6 and 8, respectively, as well as the reference processes according to Table 1 were experimentally investigated and compared. Figure 9 shows the difference between the nominal value measured inside the mold and the actual measured value of the height of the truncated cone of the test specimen disc in mm. The nominal values were determined with the before mentioned dial gauge holder directly inside the mold at 180, 60 and 20 °C mold temperature. The nominal/actual comparison at 180 °C leads to higher shrinkage in all cases, since the thermal expansion of the mold during cooling is taken into account. The nominal/actual value comparison at 20 °C and 60 °C shows no significant differences, since the mold dimension has not changed significantly in this range.



Figure 9: Measured nominal/actual difference in height of the truncated cone for the determined nominal values at 180, 60 and 20 °C mold temperature

It can be seen that the shrinkage in conventional IM cannot be sufficiently compensated, which leads to a sink mark on the components at the measuring position. As expected, in the case of conventional ICM, the shrinkage can be maintained over a longer period of time, resulting in a lower sink mark. However, the measurements also confirm the theory that support of the core by the solidifying edge area leads to a still insufficient holding pressure effect over the entire cooling time. Although the result is a slightly improved dimensional accuracy, there are still significant deviations from the nominal dimension. According to the measurements, the CIS process even allows the nominal dimension to be exceeded. This corresponds to the investigations of [33] in which the influence of pressure on the resulting component diameter was investigated. It could be shown that the component dimensions can be specifically adjusted by the CIS process. [33] Accordingly, the cycle time reduction also achieves an excess due to the high pressures, whereby the

presumably slightly inhomo-geneous temperature distribution within the component leads to shrinkage effects in the centre of the component, so that the dimension of the CIS cycle could not be completely reproduced.

The results of the photoelasticity evaluation are shown in Figure 10. Clear differences can be seen in the type of isochromatic image produced between the CIS cycles and the reference cycles. For example, the conventional IM and ICM show concentric circles around the center of the specimen, whereas the isochromats in the CIS specimens concentrate in the direction of the gate.



Figure 10: Maximum principal stress difference of the processes IM, ICM, CIS and CIS_CTO and photoelastic images in circularly polarized light

The concentric circles of the reference processes IM and ICM indicate residual stresses due to cooling. The still liquid core shrinks more than the already solidified surface layer. The component solidifies in layers from the outside to the inside, whereby stresses between the layers are frozen by the different shrinkage coefficients. Due to the geometry of the test specimen, this manifests itself in concentric circles around the center of the disk. Viewed from above, the center is free of stress and thus does not show any isochromatic layers. The photoelasticity images of the CIS samples indicate geometry-induced flow processes due to the compression stroke at the glass transition. The isochromates are concentrated in the direction of the gate, which indicates molecular orientations of the polymer chains by flowing in this direction. This falsifies the statement of the determined principal stress difference, since these are calculated in the case of CIS on the basis of orientations and in the case of the reference processes on the basis of residual stresses. Thus, the maximum principal stress difference is only permissible when comparing the processes IM and ICM or CIS and CIS_CTO. Irrespective of whether birefringence is caused by residual stresses or orientations, it has a negative effect on the optical properties in the form of disturbances of the transmitted light wave fronts. The ICM process shows slightly higher stresses than the IM process, since the compression induced stresses due to the compression of already frozen material are probably superimposed by residual stresses due to cooling. The determined stresses of the CIS samples are on a similar level. The comparsion of the photoelasticity images of the two cycles shows that the flow processes in the direction of the gate are lower at CIS_CTO, as can be seen from the lower number of isochromats. This indicates that due to the delayed heating, the gate has cooled down to such an extent that pressing melt back into the gate area is hindered by the increased viscosity of the melt in this area.

By injecting the melt into the cold cavity during the CIS_CTO cycle, a solidified surface layer will initially form, similar to the reference processes. In order to investigate any possible impairment of the microstructure impression due to the delayed heating of the mold, this was evaluated using scanning electron microscope images in comparison with the reference processes, Figure 11.



Figure 11: Scanning electron microscope images of the Fresnel microstructure for the processes IM, ICM, CIS and CIS_CTO

It can be seen that the immediate formation of a solidified surface layer in the reference processes IM and ICM leads to a deteriorated impression of the microstructure. The ICM cycle shows the worst impression, as lower injection pressures result here from injection into the partially opened cavity. The subsequent compression stroke is therefore not sufficient to mold the structure through the solidified edge layer. In contrast, in CIS, a much more precise

microstructure impression is possible by injection at a mold temperature at which the polymer is still flowable. The comparison with CIS_CTO shows that here, too, an improved impression of the microstructure is obtained compared to the reference processes. The comparison between the images of CIS and CIS_CTO indicate that the solidified surface layer becomes flowable again by heating, so that the microstructure can still be well molded by the compression stroke.

4 CONCLUSION

The investigation of the CIS cycle in comparison to the conventional manufacturing processes IM and ICM shows the advantages and disadvantages of the process. Although, compared to the IM and ICM processes, components with high dimensional accuracy and low stresses can be produced with good microstructural impressions, this is at the expense of the cycle time due to the long cooling time of the melt to solidification temperature. In order to make the CIS process more attractive from an economic point of view, a reduction of the cycle time by delayed heating of the variothermal mold was simulated in the course of the investigation. Subsequently, the experimental comparison with the conventional processes IM and ICM as well as a conventional CIS process with dimensional accuracy, regard to internal stresses and molding of microstructures was carried out, in order to validate this method.

Especially in the field of imaging optics, the conventional processes IM and ICM cannot provide satisfactory results. Here CIS represents a good alternative for the production of dimensionally stable components with low internal stresses. Furthermore, it could be shown that the previously limiting factor of long cycle times in CIS can be significantly optimized by an adapted mold temperature control. The results show that the cycle time of CIS could be reduced by up to 55 % due to a 20 s delay in the heating of the mold. Although the shrinkage is slightly higher, a positive oversize of the component could be achieved even with the cycle-time optimized process. This is in line with earlier investigations, where the component dimension can be specifically adjusted by the CIS process. The internal stresses of the CIS processes are lower than those of the reference processes IM and ICM, although it should be noted that the resulting isochromatic images differ significantly in appearance. The reason for this could be that the isochromatic images of the IM and ICM processes are based on residual stresses, whereas the images of the CIS samples are based on orientations caused by geometry-induced flow processes at the glass transition. The microstructure impression is hindered in the conventional processes IM and ICM due to the formation of a solidified surface layer. Due to the high mold temperature during the CIS process, the flowability of the melt is maintained, which is reflected in an improved microstructure impression. Here, too, the cycle-time-optimized variant shows hardly any disadvantages compared to the conventional process. Thus, the cycle time reduction determined by simulation

could be validated by experimental comparison within the scope of the investigation. Current investigations are concerned with the compensation of temperature differences due to high pressures and the associated further possible reduction of the cycle time.

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Appendix

Temperature-dependent specific heat capacity:

Temperature [°C]	Specific heat capacity	Heating/Cooling Pate [K/a]
	[J/kg*K]	
32	1200	
80	1433	
100	1522	
125	1647	
129	1677	
133	1720	
137	1774	
141	1845	-0,333
148	1939	
160	1982	
190	2054	
217	2107	
251	2177	
280	2215	
315	2247	

Temperature [°C]	Thermal conductivity [W/m*K]
38.7	0.2007
48	0.1958
67.9	0.1947
88	0.1969
108.1	0.1985
128.7	0.2206
148.4	0.2531
168.1	0.2476
187.3	0.2553
207	0.2478
226.6	0.2532
246.8	0.2477
267	0.2546
287.5	0.2581
307.5	0.2591

Material Data: Temperature-dependent Thermal conductivity:

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