High-performance Compounds. Low-density injection moldable thermoplastics can save resources and costs in lightweight construction applications. By incorporating hollow glass beads in matrix polymers such as PEEK and polyamide 6, it has been possible to produce technically sophisticated compounds. These can be used, for example, in aviation, automotive engineering, and medical technology.

A new compound with the trade name Tecacomp LW (low weight) comprises the thermoplastic polyetheretherketone (PEEK) or polyamide 6 (PA6) and up to 40 wt.-% hollow glass microspheres. It has a density of less than 1.0 g/cm³. In comparison with previously used, glass-fiber-reinforced grades, the new material offers weight savings of up to 40 % and price advantages.

Further development of this lightweight construction material requires demonstrating its processability on injection molding machines. It is essential for the hollow glass microspheres to withstand high pressure if they are to be suitable for melt processing. At the same time, they must be resistant to high temperatures so that they can be incorporated in polymers with a high melting point, such as PEEK. The hollow glass microspheres marketed under the trade name 3M Glass Bubbles iM30K from 3M Deutschland GmbH in Neuss, Germany, can be used without any problem for this purpose. They have extremely high isostatic compressive strength up to 2,000 bar. Their nominal density is 0.60 g/cm³. They are heat-resistant up to 600°C, which enables them to be used in high-temperature plastics.

In addition to the right filler, a specially designed compounding technique was required, e.g. to ensure filler survival and allow high volume filler loading. This was achieved through a special process modification developed by Ensinger GmbH & Co. in Nufringen, Germany.

Formulation and Process Development

The model substances chosen for the development work were the engineering plastic, PA6, and the high-performance polymer, PEEK. The first step was to modify the compounding process so that the microspheres could be incorporated into the polymer matrix without damage. For this purpose, a series of compounds with filler contents of up to 40 wt.-% and 60 vol.-% were prepared. These trial compounds were then characterized with respect to their density, mechanical properties (tensile and flexural) and thermal behavior (linear thermal expansion and shrinkage). A morphological study was also carried out by scanning electron microscopy (SEM).

An important step in formulation development was optimization of the filler-matrix bond by applying a special coating to the hollow glass microspheres (Fig. 1.). For compounding, the screw configuration and feed system were redesigned to create optimum shearing conditions in the compounder. The shearing process was required to disperse the microspheres homogeneously in the poly-
mer without damaging them. At the same time, in order to achieve high volume filler loading in the compound, special measures were necessary in terms of feed technology and venting, since the low density of the hollow glass microspheres results in low bulk density and fluidizing behavior.

The measured density values of the model substances Tecacomp PA6 LW and Tecacomp PEEK LW, determined as relative density according to DIN EN ISO 1183, show only slight deviations from the theoretical values. Both incorporation of the hollow glass microspheres in the compounding process and injection molding of the filled polymer were accomplished without destruction of the microspheres. With the material containing 40 wt.-% hollow glass microspheres, it is possible to achieve a density reduction of 24 % over the value for the unfilled polymer. Compared to compounds filled with solid glass microspheres or glass fibers, the weight advantage is 43 %. As a result of these weight savings, volume-based material costs can also be reduced (Fig. 2).

**Mechanically and Thermally Stable**

In tensile tests according to DIN EN ISO 527, increased tensile modulus and tensile strength were demonstrated. The hollow glass microspheres also increased modulus and strength values in the flexural test (DIN EN ISO 178). So the microspheres have a stiffening, reinforcing effect in the compounds (Fig. 3).

In addition, thanks to the use of hollow glass microspheres, the compound shrinks far less on cooling, and so makes it possible to produce components that are largely warpage-free. Owing to the spherical shape of the hollow glass beads used as a filler, uniform shrinkage is achieved in all spatial directions. Testing based on DIN EN ISO 294-4 showed reduced shrinkage compared with the unfilled material and isotropic shrinkage in contrast to fiber-reinforced material (Fig. 4).

Thermal expansion, tested by thermomechanical analysis, was also considerably reduced by the hollow glass microspheres. The pre-requisite for this is high volume filler loading. This can be achieved by the special filler and compounding know-how (Fig. 5).

A key requirement ensuring the advantageous property profile of these compounds is a good bond between the filler and polymer matrix. By adjusting the microsphere coating to suit the particular polymer matrix, product properties were further optimized. Thanks to the good filler-polymer adhesion so achieved, it was possible to stop the microspheres break-

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**Fig. 1. SEM of hollow glass microspheres (photo: 3M)**

**Fig. 2. A comparison of the density values measured on standard tensile test bars made from PA6 compound with the theoretical values shows the stability of the hollow glass microspheres in the compounding and injection molding process (source: Ensinger)**

**Fig. 3. The values from tensile and flexural testing of PA6 compounds show increased strength and stiffness as a result of the hollow glass microspheres (source: Ensinger)**

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**Fig. 4.**

**Fig. 5.**
ing out under mechanical stress and so prevent premature component failure. The scanning electron micrograph shows the cryogenic fracture surface of a PEEK compound with specially coated hollow glass microspheres. These are completely wetted by the polymer and well integrated into the matrix. The morphology of the compound correlates with its mechanical properties – through the use of microspheres with an optimized coating, the toughness and strength of the compound are considerably increased (Figs. 6 and 7).

Properties and Application Potential

The combination of heat resistance, lightweight, and favorable mechanical properties offered by these new materials opens up applications in aviation (Title picture), transport, and the automotive industry. The main advantage of these compounds is their light weight, which enables them to achieve resource and cost savings. Thanks to the stiffening function of the hollow glass microspheres, the reduced thermal expansion and the thermal stability of the base polymers, these compounds are suitable for components exposed to thermal and mechanical stress in service. On account of their low warpage tendency, they are also very suitable for medical technology products subjected to frequent temperature changes, e.g., sterilization containers. The reduced molding shrinkage, isotropic properties and volume-related cost advantages of the compounds are other positive benefits for injection molders.

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