Conditioning Ultramid[®] moldings

Technical information



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Ultramid® (PA)

BASF's Ultramid[®] grades are molding compounds on the basis of PA6, PA66, various copolyamides such as PA66/6 and partially aromatic polyamide. Ultramid[®] stands out for its high mechanical strength, stiffness and thermal stability. Moreover, it is tough at low temperatures, has a favorable sliding friction behavior and permits problem-free processing. Owing to its excellent properties, this material has become indispensable in almost every technical realm for a lot of components and machine elements, also as high-grade electric insulating material and for many special applications.

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Conditioning Ultramid® moldings

General

Polyamide (PA) moldings are completely dry after they have been produced. If they are exposed to moist air or immersed in water, they absorb moisture at a rate that depends on the conditions. If they are exposed to accelerated conditions, referred to as conditioning, they can attain the desired moisture content in the shortest possible time, and their properties, e.g. the impact strength, can thus be improved.

Polyamide 6, polyamide 66, and copolyamide 66/6 absorb comparatively large amounts of water and must therefore be conditioned. However, conditioning has very little effect on freshly injection-molded Ultramid[®] T parts (Fig. 1) and can thus be dispensed with. Under these circumstances, it is only in exceptional cases, e.g. the need to meet certain dimensional specifications, that there would be any point in conditioning.

The aim of conditioning dry polyamide parts is to allow them to absorb moisture as rapidly as possible. Standard practice is to condition the articles to the equilibrium moisture content in a standard laboratory atmosphere of 23 °C and 50% relative humidity. They can also be conditioned to the equilibrium moisture content in other climates defined by a given temperature and relative humidity.

The maximum moisture content is not attained unless the part is permanently immersed in water, but only the equilibrium moisture content at 23°C and 50% relative humidity is of significance in practice.

Why conditioning?

The absorption of water gives rise to changes in the properties and dimensions of dry polyamide parts. If the absorption takes place under service conditions, the changes may have an adverse effect in many applications. For this reason, Ultramid[®] parts that are to be subjected to strong elastic deformation and high impact loads should display their characteristic high impact strength from the very outset (Fig. 2).

Another reason for conditioning is that the dimensions, strength, and rigidity of many engineering parts are specified within narrow tolerances. This entails that their moisture content must roughly correspond to that in the equilibrium state under the climatic conditions concerned (Table 1).

Under normal conditions, Ultramid[®] parts absorb moisture very slowly from the air, particularly if they have thick walls (Fig. 3). Hence, if they are not conditioned, it would take a very long time before the equilibrium moisture content and thus the final dimensions could be attained. On the other hand, if the parts were to be conditioned, e. g. by storage in a humid room at 40 °C and 90% relative humidity or by immersion in water at, say, 40 °C, the equilibrium moisture content could be attained in a short period of time (Fig. 4).

Since conditioning involves a certain outlay, it is necessary only if polyamide's great toughness or dimensional stability has to be exploited from the very beginning. This also applies to the glass-reinforced grades.



Fig. 1: Equilibrium moisture content of Ultramid[®] as a function of the relative humidity in the 10-70 °C temperature range (scattering $\pm 0.2-0.4$ absolute).



Fig. 2: Increase in impact strength brought about by absorption of moisture during conditioning. Determined by the ISO 179/1eA notched impact test. The specimens were of 4 mm thickness. They were immersed in water at 23 °C and subsequently kept for five days at 23 °C and 50 % relative humidity until the equilibrium moisture content was attained throughout.



Fig. 3: Moisture absorption by unreinforced Ultramid[®] in a standard atmosphere of 23°C and 50% relative humidity as a function of time for various wall thicknesses.



Fig. 4: Water absorption by unreinforced Ultramid[®] on immersion in water at 40 °C or in a humid room at 40 °C and 90% relative humidity as a function of time for various wall thicknesses.

Atmosphere	Thickness	Ultramid [®] A (PA66)				Ultramid® B (PA6)			
	of specimen	unrein	unreinforced 30% GF reinforcement		unreinforced		30% GF reinforcement		
		Minimum % wt	Maximum % wt	Minimum % wt	Maximum % wt	Minimum % wt	Maximum % wt	Minimum % wt	Maximum % wt
Offices Workshops Ambient air	4 mm 4 mm 4 mm	1.0 1.3 2.3	2.2 2.3 4.6	0.7 0.9 4.6	1.5 1.6 3.2	0.8 1.2 2.0	2.3 2.7 5.2	0.56 0.9 1.4	1.6 1.9 3.6
Offices Workshops Ambient air	8 mm 8 mm 8 mm	1.5 1.7 2.2	1.9 2.1 2.9	1.0 1.2 1.5	1.3 1.5 2.0	1.7 1.9 2.7	2.1 2.3 2.4	1.2 1.3 1.9	1.5 1.6 2.4
Standard atmosphere 23°C/50% H _R saturation	-	2.8 ± 0.3		1.8 ± 0.2		3.0 ± 0.4		1.8 ± 0.2	

* The moisture content of a reinforced grade can be derived from the value for an unreinforced grade by taking into account the glass-fibre content. For example, grades with 30% glass fibres contain only 70% of the moisture of unreinforced grades. The moisture content of 25% glass-filled grades is 75% of the value for unreinforced material.

Table 1: Moisture content in various atmospheres - empirical values

Procedures for conditioning

Good procedure for conditioning are to immerse the parts in hot water and to expose them to a warm, humid climate or to saturated steam.

Conditioning in hot water is comparatively simple because it necessitates only a small outlay in equipment. Best results are obtained in a water bath thermostatically controlled in the 40-90°C range. The bath must be well insulated and covered to avoid excessive heat losses. If the surface of the water is covered with, say, table-tennis balls, heat losses can be largely avoided. A disadvantage of the water immersion method is that stains and impairment of the finish cannot always be avoided, particularly in glass-reinforced, coloured polyamide moldings.

Particularly mild conditions exist if Ultramid[®] is conditioned in a humid atmosphere at a temperature of up to about 40 °C and a relative humidity higher than 80%. This method avoids the disadvantage mentioned above and can be carried out with commercial conditioning equipment. If large parts, e.g. housings and tanks, have to be conditioned, it would be advisable to equip a readily accessible conditioning room with the necessary heating and hygrothermostatic control.

The simplest means of conditioning without any outlay on equipment consists of keeping the Ultramid[®] parts in waterproof PE sacks with a wall thickness greater than 0.1 mm and containing 5-10% of water, expressed in terms of the weight of the parts. The higher the temperature at which the parts are kept and the thinner they are, the shorter the time required for conditioning. An idea of the time required can be obtained from the conditioning diagrams shown in Figs. 5-8.

If Ultramid[®] machinery parts have very thick walls, they can be conditioned in saturated steam at temperatures of up to about 120°C. The temperature limit of 120°C should not be exceeded, as otherwise the polyamide may be damaged after only a few hours.



Fig. 5: Time required for conditioning unreinforced Ultramid[®] B as a function of the wall thickness and the water bath temperature.



Fig. 6: Time required for conditioning glass-reinforced Ultramid[®] B as a function of the wall thickness and the water bath temperature.



Fig. 7: Time required for conditioning unreinforced Ultramid[®] A as a function of the wall thickness and the water bath temperature.



temperature.

Storage after conditioning

The amount of moisture absorbed by an Ultramid® part after conditioning in accordance with the recommendations given above can be found by weighing. However, the figure obtained does not yield any information on the distribution of moisture over the cross-section of the part. The aim is uniform distribution of moisture over the entire cross-section within a short period after the part has been removed from the conditioning bath. However, this can be realized only if the wall thickness is less than about 3 mm. If it is greater, a significantly higher moisture content will result at the surface, and the centre will remain almost dry. If parts of this nature are kept in air after they have been conditioned, some of the water from the almost saturated layer at the surface will be released again into the air, and another portion of the excess will penetrate further into the almost dry core. Experiments have revealed that as much as 50 % of the water absorbed on conditioning thick parts may remigrate outwards. For this reason, it is advisable to pack Ultramid® moldings in a humid atmosphere after they have been conditioned, e.g. in PE bags. A slight release of water from the saturated layer at the surface is of no significance; a moisture content of about 2.5 % in Ultramid® A and B suffices in most cases. This figure corresponds roughly to an equilibrium moisture content at 23 °C and a relative humidity of 40-50 %.

Determination of the time required for conditioning

By the law of diffusion

The rate of water absorbed by Ultramid[®] parts roughly follows a simplified form of the law of diffusion, i.e.

$$t = \frac{C_t^2}{C_s^2} \cdot \frac{1}{2.256^2} \cdot s^2 \cdot \frac{1}{D}$$

where c_t is the water content in % at time t, c_s is the water content in % at saturation, c_t/c_s is the degree of saturation, D is the diffusion coefficient in cm²/s, s is the wall thickness in mm. The factor 2.256 depends on the geometry of the part (in this case, panels).

The diffusion coefficient depends on the PA grade (Table 2) and increases rapidly with temperature, the reason for the high water absorption at elevated temperatures. As is evident from the equation, the time t required for conditioning to a given water content increases with the square of the wall thickness. Thus, if 18 hours are required for a thickness of 1.5 mm, four times that time, i.e. 72 hours, would be required for 3 mm thickness under the same climatic conditions to attain the same water content in the same material.

By conditioning diagrams and nomogram

The time required for conditioning Ultramid[®] A and B (PA66 and PA6) as a function of wall thickness and temperature can be derived from Figs. 5 to 8 for the case of permanent immersion in aqueous media. These diagrams are applicable for immersion in water in most cases. The nomogram in Fig. 9 is also useful, particularly in cases in which the diagrams for the determination of the time required for conditioning no longer suffice.

Example: Ultramid[®] A3K injection molding of medium wall thickness s = 10 mm is to be conditioned in water at 90°C to a water content of c_t \simeq 2.3 %. Determine the time required for conditioning.

Solution: In Fig. 9a, draw a straight line from the waterbath temperature (90°C) to the wall thickness (10 mm) to intersect the pivot line at the point P. Connect the desired moisture content (2.3 % on the scale to the left of the diagram), and extend this straight line to intersect the time scale to give the time required for conditioning (20 hours).

Ultramid®	c _t (%) equilibrium moisture content at 23°C/50% H _R	$c_s^{}$ (%) water content at saturation DIN EN ISO 62		Coefficient of diffusion D · 10 ⁻⁸ (cm ² /s) at a water temperature of					
		<80°C	>80°C	20°C	40°C	60°C	80°C	100°C	120°C
B unreinforced with	3.0	8.5 to 9	10	0.4	1.5	5.5	20	55	150
glassfibers (30%)	2.1	5.8	6	0.4	1.5	5.5	20	55	150
A unreinforced with	2.5 to 2.8	7.5 to 8	9	0.2	0.9	3.5	12	35	90
glassfibers (30%)	1.7	5	5.5	0.2	0.9	3.5	12	35	90
T unreinforced with	1.6 to 2.0	6.5 to 7.5							
glassfibers (30%)	0.6 to 1.0	4 to 5							

Table 2: Conditioning of Ultramid[®] panels. Determination of the time required for conditioning in water with the aid of the coefficient of diffusion (D).

Key to the use of the nomogram: 1. Draw a straight line from the given water bath temperature to the given wall thickness

- → Point P (= intersection with the pivot line)
- 2. Draw a straight line from the desired moisture content to the Point P and extent it to the time scale

 time required for conditioning

Ultramid® A



Fig. 9a: Nomogram for the determination of water absorption by Ultramid[®] A in accordance with the law of diffusion (the example given on page 9 has been entered in the diagram).

Ultramid® B



Fig. 9b: Nomogram for the determination of the water absorption by Ultramid® B in accordance with the law of diffusion.

The behavior of conditioned moldings under normal seasonal climatic fluctuation

When the climatic conditions change, conditioned articles release their moisture content just as slowly as they absorbed it in the dry, freshly molded state. In other word, the effect of fluctuations in atmospheric humidity on the moisture content of the moldings is insignificant and subject to a considerable lag.

Since the associated change in the linear dimensions is less than this slight fluctuation in moisture content by a factor of 3, the effect of fluctuations in climatic conditions is usually negligible in practice.

Behaviour at low temperatures

Even at low temperatures and the associated low absolute atmospheric humidity, Ultramid[®] parts do not tend to dry out and become brittle. The reason for this is that the lower the temperature, the less the rate of moisture release. Experiments along these lines yielded the values listed in Table 3.

Ultramid [®] unreinforced	А	В
Initial value	2.7	3.0
at 20°C after 30 days dry storage	1.7	1.9
at -25°C after 30 days dry storage	2.4	2.3

Table 3: Moisture content (%) of Ultramid[®] moldings of 1 mm wall thickness

The rate at which water diffuses through PA falls rapidly with decreasing temperature. Consequently, absorbed water is released only very slowly, despite low atmospheric humidity. This is why Ultramid[®] parts retain their toughness in cold climates, even when outdoors. The plasticizing action of the water increases the impact strength of the polyamide and remains effective even at subzero temperatures. It is often erroneously assumed that the water absorbed in the polyamide "freezes" and thus embrittles the moldings, but this is not the case.

Dimensional changes caused by the absorption of water

The fact that the length of a molding changes on the absorption of water must be allowed for in design. The change is much less in the reinforced than in the unreinforced grades. On average, it is about one-quarter of the percentage increase in weight of the unreinforced products. Thus, if 2 % wt of water is absorbed, the increase in length is about 0.5 %. The change in length of the reinforced grades depends on the orientation of the fibres. In the direction of the fibres, the increase in length is only one-quarter of that in the unreinforced grades.

Potential adverse effects of conditioning and their avoidance

Inhibition of corrosion in injection moldings with steel cores

It can usually be expected that steel cores in injection moldings will corrode during conditioning. If the moldings are conditioned with hot water or steam, corrosion can be prevented by adding certain basic substance, e.g. 0.2-0.5 % of hexamethylenetetramine. In this case, there is no fear of damaging the Ultramid[®].

Discoloration

In common with any other form of heat treatment, conditioning may discolour pale coloured moldings. The intensity of the discoloration – generally yellowing – depends on the duration and temperature of exposure and on the processing conditions. Molding compounds that have been thermooxidatively degraded or contain recycled material have a particularly strong tendency to discolour during conditioning. The intensity of the discoloration may be increased by particles of iron, e.g. that introduced by wear of the rotors during the size reduction of scrap. Under certain circumstances, Ultramid[®] A may even discolour to pink shades.

Discoloration can be completely avoided by adding 0.2 to 1 % of sodium bisulfite to the water in the conditioning bath, which should be of drinking water quality and largely free from traces of heavy metals. Sodium bisulfite is a compound that is readily available, does not harm Ultramid[®], and is easy to handle. It also allows discoloured articles to be decolorized again. For this purpose, a bath temperature of about 80 °C and an immersion period of one to three days are generally required.

Sodium bisulfite solutions may cause annoyance owing to their inherent, faint sulfur dioxide odour, and they may attack the materials of the vessels in which they are contained. In the latter case, resistance of the material should be verified beforehand.

Avoidance of white deposits on conditioning Ultramid[®] B parts

Ultramid[®] B contains small amounts of low-molecular-weight fractions. On immersion in hot water, these fractions may migrate to the surface of the molding and occasionally form white stains. These deposits are readily soluble in aqueous alcohols and can thus be easily removed, e.g. with ethanol or isopropanol. If objections exist against cleaning with alcohol, the deposits can be avoided at the outset by conditioning the moldings in a moderately warm, humid atmosphere (temperature <60 °C) instead of in hot water.

Excessively hard water and high temperatures may give rise to scale deposits. They can also be avoided by lowering the temperature or softening the water.

Effects of error in conditioning

Plane surfaces, e.g. on housings, frequently distort on conditioning in water and a humid climate, even at moderate temperatures. The reason for this is that the softening effect caused by the absorption of moisture may relieve existing molded-in stresses. The difficulty can be partly overcome by careful design, particularly of the gating, and the correct choice of mold temperature. Warpage can often be avoided by conditioning at room temperature or with moderate heat to a low moisture content, e.g. 1 to 1.5 %. Glass-reinforced grades tend to warp more than unreinforced ones.

During conditioning, cracks may form along poorly fused weld lines. The risk applies to all moldings if the injection and mold temperatures were too low. If this effect occurs, it is advisable to check the efficacy of the processing parameters by examining the structure on the fault under a polarizing microscope.

For your notes

Selected Product Literature for Ultramid®:

- Ultramid[®] Product Brochure
- Ultramid[®] Product Range
- Ultramid[®], Ultradur[®] and Ultraform[®] Resistance to Chemicals

Note

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