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Impact strength of nylon 6 and 66 in the dry and moist states

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The impact strength of nylon was determined by the modified Charpy test with double-V notches described in DIN 53 753, because it cannot be reliably assessed by the notched Charpy and Izod tests. This test was adjusted to obtain a sensitive, accurate and reproducible method for the determination of impact strength in the dry and moist states. In the light of the criterion thus gained, it is shown that the dry "as-moulded" state is unstable and that the impact strength decreases exponentially with storage time. Therefore, comparison tests are not valid unless the measurements are performed within a restricted period of time after the specimens had been demoulded. If the age of the specimens is unknown, a reproducible "as-moulded" state can be regenerated by an annealing procedure. The state of "conventional" moisture content that is taken as a reference for comparing data obtained on moist nylon is the equilibrium moisture content in a standard laboratory atmosphere. Various methods are discussed for attaining this state rapidly by accelerated conditioning. An atmosphere of 70° C and 62% relative humidity allows accurate adjustment to the "conventional" moisture content but does not result in a stable reference state; for instance, the impact strength decreases within several weeks after conditioning.

1. Introduction

Polyamides have ideal properties for parts subjected to high mechanical stresses. The most important for many applications are the rigidity, strength and toughness.

The main factors that govern the properties are the chemical structure and the viscosity number, but a measurable change of the toughness, for example, may be effected by compounding and coloration. This explains the long-standing wish for test methods that allow the toughness of nylon moulding materials to be checked on a routine basis.

Normally, the notched impact strength is cited as the criterion for this purpose in various publications, data collections and specifications. Apart from the fact that one-point measurements of this kind are of a dubious nature, it has been found that this test is not very suitable for nylon, because it does not allow sufficient differentiation and a high factor of uncertainty is involved in the measurements. Nevertheless, by judicious modification of the test conditions and the method of preparing the specimens, the notched impact test has been converted into a sensitive method that will also yield reliable results for polyamides.

If reproducible results are to be obtained, the state of the specimens must be defined. Since the properties of unmodified nylon 6 and nylon 66 greatly depend on the moisture content, the dry state of the specimens is usually required to ensure reproducible results. In publications, standards and specifications it is customary to describe the dry state as "freshly injection moulded". The results obtained in this study with a modified impact test revealed for the first time that this description did not adequately apply to a defined, dry state for unmodified nylon 6 and nylon 66 specimens. However, inadequately defined specimens can be converted into a definitely defined state by programmed conditioning.

It is alleged that if the properties of nylon are measured on specimens in the dry state and the results are arranged in the order of their magnitude, the same sequence will apply to the properties of specimens in a moist condition. In order to check this thesis, a means for adjustment of a defined moisture content, the moist reference state, are required. New methods were studied for attaining a reference level by accelerated conditioning. By means of the modified impact test it was demonstrated that the reproducibility of a defined moist state is still insufficient, even if it represents the "conventional" moisture content in equilibrium with the standard laboratory atmosphere of 23° C and 50% relative humidity (r.h.).

Data for specifications, publications, etc, must be presented on a basis that allows comparison. In this respect, the studies show that special boundary conditions must be observed for unmodified nylon 6 and nylon 66 and that these conditions also apply to the quality control of mouldings.

2. Impact strength of specimens in the dry state

Any test method for determining the toughness of dry specimens must allow the products concerned to be classified according to their behaviour in practice. It

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must also be accurate enough to permit a comparison of quality and should yield results that are reproducible in the sense of ISO 3534. This entails that the specimens must be in a comparable state, a demand that is not easy to fulfil for nylon 6 and nylon 66.

In the tensile test, the figures obtained for the elongation at break of these moulding materials are subject to considerable scatter. Hence the corresponding values for the failure energy are unreliable. For this reason, an investigation was made to determine whether the Charpy impact test would yield better results.

For various reasons, the notched impact test with a U-notch according to DIN 53 453 and ISO 179/2C is unsuitable for the quality control of dry nylon. The notch is of indeterminate radius ($r < 0.1$ mm) and cuts through the outer fibres, which are so important for the impact resistance. However, the main point of this test is that the stress rises so rapidly at the base of the notch that relaxation, which governs the toughness, is almost completely suppressed. Consequently, a realistic differentiation of various qualities is impossible.

These disadvantages can be avoided in the DIN 53 753 test with double-V notch (see Fig. 1). Since the notches are located at the sides of the specimen, the initial crack is formed in the undamaged outer fibres. The notch radius can be adjusted so that the time to effect failure [1] is an optimum for the moulding compound to be tested [2].

The following products with normal and rapid solidification rates (normal-setting, rapid-setting or nucleated) and in the same range of viscosity number were taken for the purpose of determining the toughness of dry and moist polyamides:

ISO 1874–PA 6, MHNR*, 14-030

ISO 1874–PA 6, MHNR, 14-030 N

ISO 1874–PA 66, MHNR, 14-030

ISO 1874–PA 66, MHNR, 14-030 N

Random samples were taken to ensure that the difference in behaviour of unreinforced products in other ranges of viscosity number was only slight.

The first step consisted of determining whether DIN 53 753 would apply, with test speicmens cut out of the uniaxially oriented rectangular plate [2, 3] as described in ISO 2557/2. The effect of anisotropy on this plate was studied at the same time. In order to simulate as closely as possible the distribution of amorphous and crystalline phases that is encountered in practice, a figure of 2 mm was laid down for the thickness h of the platelets, which were injection-moulded under the conditions quoted in ISO 1874/2. By means of a multiple-blade saw, the platelets were cut up into bar specimens parallel and perpendicular to the direction of flow. This is illustrated in Fig. 2.

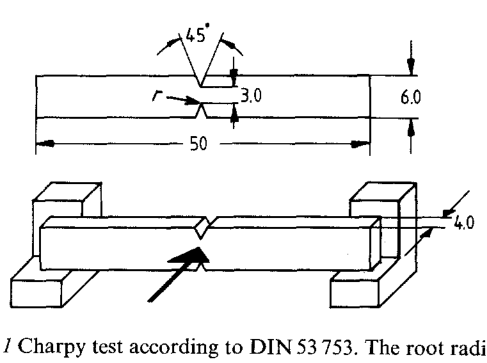

Figure 1 Charpy test according to DIN 53 753. The root radius r of the double-V notches can be adapted to the material to between 0.1 and 3.0 mm. Dimensions given in mm.

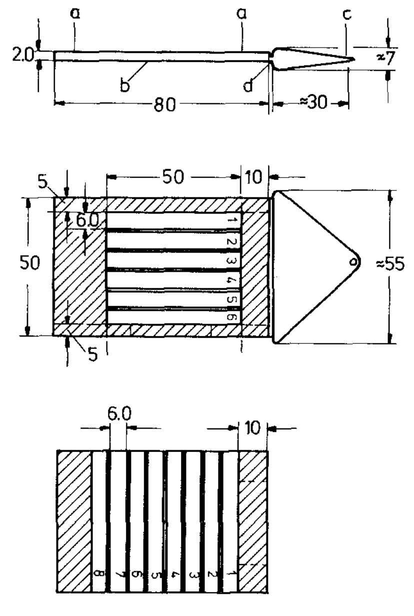

Figure 2 Unaxially oriented specimen panel according to ISO 2557/2 [2, 3]; dimenions in mm (above) and cross-sections parallel (centre) and perpendicular (below) to the direction of flow: (a) ejector, (b) side tested, (c) sprue, (d) film gate (thickness 1.0 ± 0.1 mm).

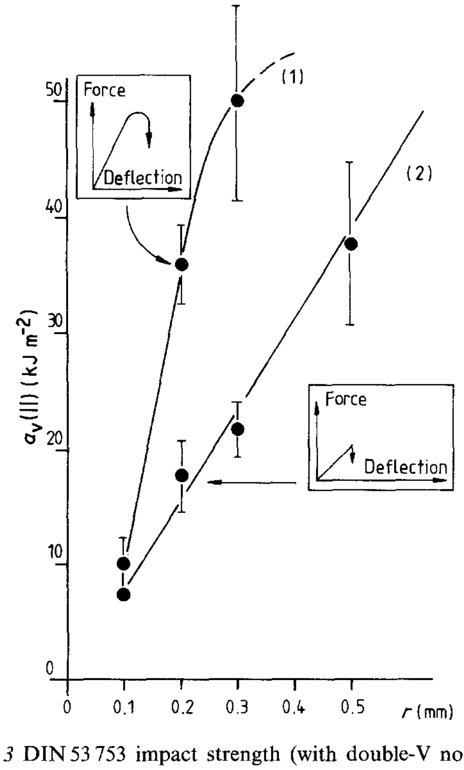

Figure 3 DIN 53 753 impact strength (with double-V notch) as a function of notch radius; specimens taken from panels parallel to the direction of flow as shown in Fig. 2. (1) Nylon 6 solidifying at a normal rate, (2) nylon 6 nucleated. State: dry.

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*M = intended for injection moulding, H = heat-stabilized, N = uncoloured, R = contains mould release additive.

Double-V notches of various radii were cut into the 50 mm × 6 mm × 2 mm specimens thus obtained, which were kept in a desiccator until they were required for testing.

Fig. 3 shows typical results obtained in this series of experiments on nucleated and normal-setting dry polyamides. At a notch radius of $r = 0.1$ mm, these results are similar to those obtained with the u-notch, i.e. the relaxation is almost completely suppressed. As a result, the widely differing products cannot be differentiated from one another here. As the notch radius increases and the time required for failure becomes longer, the failure energy for the various products rises at different rates. At a value of only $r = 0.2$ mm, the impact strength of the normal-setting moulding material is already twice as high as that of the nucleated (rapid-setting) moulding material. At radii of 0.4 mm upwards, complete failure no longer occurs in the normal-setting moulding material.

The nucleated nylon 6 moulding material was taken here as an example to demonstrate the effect of anisotropy. The curve obtained is shown in Fig. 4. As is to be expected, no difference is apparent until the notch radius increases. Afterwards in contrast to the results obtained with amorphous thermoplastics [2, 3], the impact strength of specimens taken at right-angles to the direction of flow is higher than that of specimens taken parallel to the direction of flow. This applies to all the polyamides investigated here. The degree of anisotropy in normal-setting products can be changed somewhat by altering the injection-moulding conditions, but the direction of the anisotropy is the same for all the products and injection-moulding conditions investigated.

The results confirmed that the Charpy impact test with a double-V notch is fundamentally suitable for comparing the qualities of different types of nylon. If the notch radius is large enough, the measurements on specimens in the dry state reflect the behaviour of the products investigated in practice.

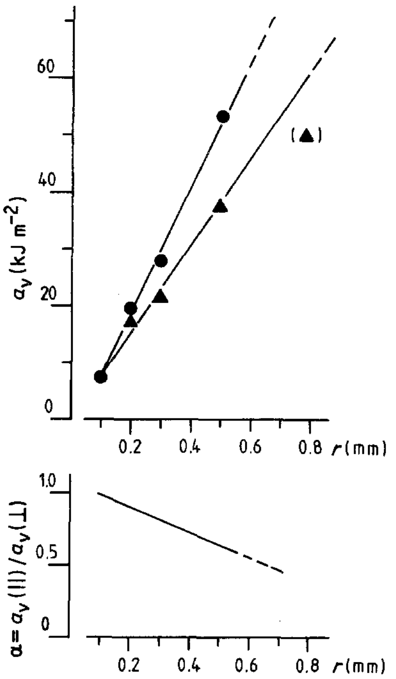

Figure 4 Anisotropy of DIN 53 753 impact strength as a function of the notch radius measured on specimens taken (▲) parallel and (●) perpendicular to the flow from the panels shown in Fig. 2. Moulding compound: nylon 6 nucleated (dry).

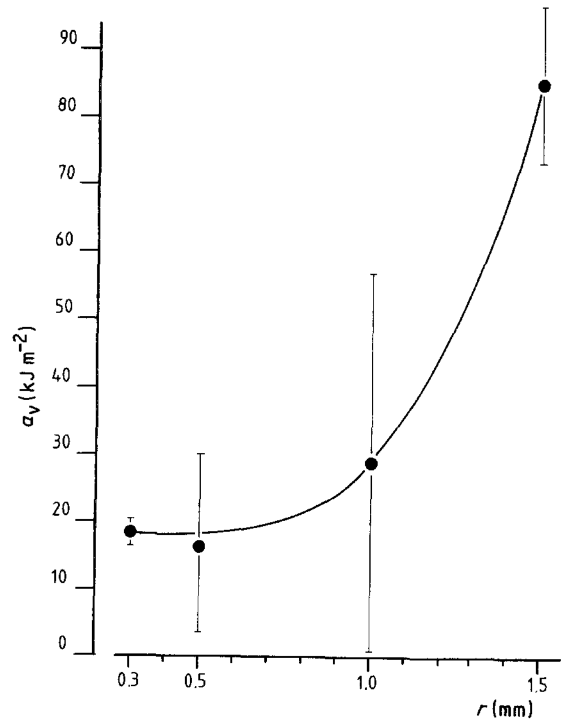

Figure 5 DIN 53 753 impact strength as a function of notch radius as measured on 50 mm × 6 mm × 4 mm standard bar specimens of normal-setting nylon 6.

In order to apply the principle underlying this test method to a standardized specimen dimension, ISO 179 Type 2 (50 mm × 6 mm × 4 mm), another optimum size had to be found for the notch radius. The reason for this is that doubling the specimen thickness entails, as a first approximation, a twofold increase in the rate of deformation, and this must be compensated by enlarging the notch radius.

The results of the first measurements were disappointing and are shown in Fig. 5. The example taken was a normal-setting nylon 6. As would be expected, the thicker specimens do not allow differentiation until the notch radius is more than

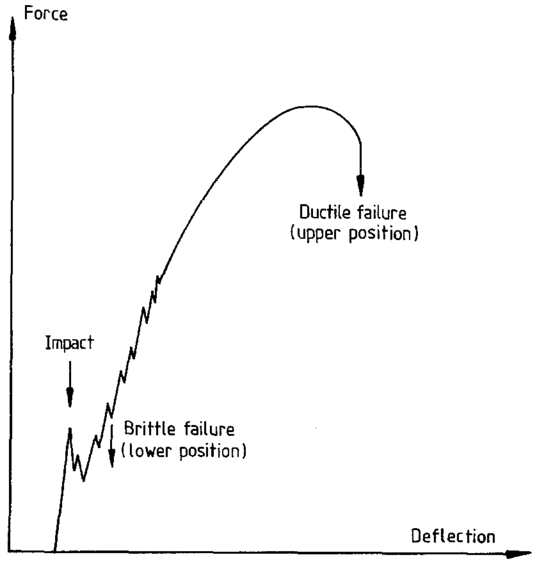

Figure 6 Bimodal failure of dry nylon in the DIN 53 753 impact test on specimens with a notch radius of $r = 0.5$ to 1.0 mm.

TABLE I Impact strength a_v (DIN 53 753) for PA 6, MHNR, 14-030 (dry), specimens ISO 179 Type 2 ($n = 20$)

Notch radius (mm)	Ductile fracture		Brittle fracture	
	%	a_v (kJ m^{-2})	%	a_v (kJ m^{-2})
0.3	100	18.6 ± 2.5	0	–
0.5	50	29.1 ± 5.3	50	3.8 ± 2.9
1.0	45	57.8 ± 5.2	55	4.7 ± 2.1
1.5	100	83.4 ± 11.6	0	–

0.5 mm, but the shape of the curve is entirely different from that in Fig. 3. The accuracy of the mean values (variation coefficients > 50%) with notches of medium radii precludes any statistical significance being given to the test results.

The reason for the large scatter in the experimental results is a bimodal distribution for the fracture mechanism. If the pendulum is fitted with an electronic recording device, it can be demonstrated that some of the specimens already show brittle fracture during the vibrations that follow the impact (Fig. 6). Failure is not initiated in the outer fibres at the base of the notch but within the body of the specimen. This is evident from the appearance of the planes of fracture.

Individual values for the impact strength, broken down into the figures for ductile and brittle fracture, are listed in Table I.

Within both failure categories, the scatter of the experimental results is quite normal. If the figures for the ductile fracture are plotted against the notch radius, the shape of the curve obtained (Fig. 7) is similar to that shown in Fig. 3, as was originally expected.

The proportion of failures accounted for by brittle fracture in the products investigated (see above) varies somewhat but cannot be significantly changed by altering the injection-moulding conditions. It has been seen that no differentiation is possible with a notch radius of $r \leqslant 0.3$ mm and that all the specimens of the tougher unreinforced nylon resins do not completely fail at $r \geqslant 1.8$ mm. Consequently, if the quality control for nylon 6 and 66 by the DIN 53 753 notched-impact test is to be performed on specimens in the dry state and with the standardized specimen dimensions of 50 mm × 6 mm × 4 mm, the only notch radius that can be considered is $r = 1.5$ mm. Even then, up to 5 to 10% of the specimens may still occasionally undergo brittle fracture.

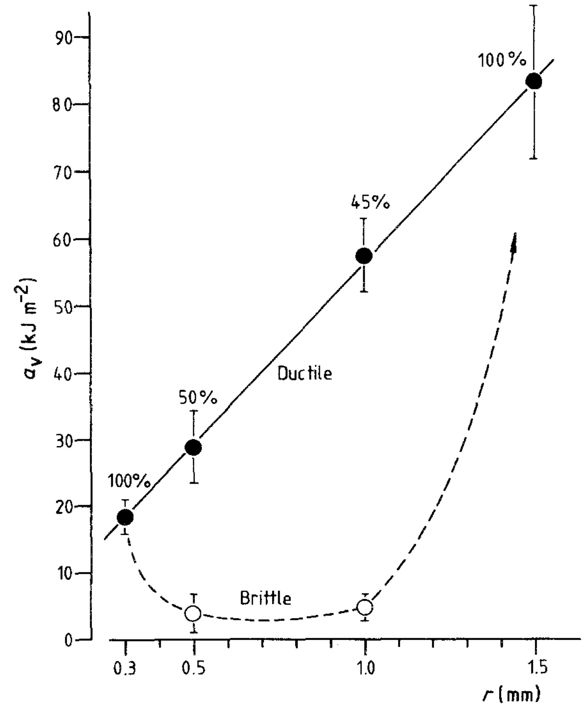

Figure 7 Test series represented in Fig. 4 separated into (●) ductile and (○) brittle failure; the percentage ductile failures are quoted in the diagram ($n = 20$).

With this notch radius, the ratio of the impact strength a_v of the normally solidifying nylon 6 to that of the nucleated nylon 6 is again roughly 1 : 2, as was already demonstrated above with test specimens of 2 mm thickness (see Fig. 3).

3. Increasing the reliability of the measurements

The accuracy of typical series of measurements by the Charpy tests mentioned above, i.e. variation coefficients of 10 to 20%, is inadequate for more accurate investigations, e.g. in determining manufacturing tolerances for quality control. Milling the notches can be eliminated as a source of error in this case, because for the purpose of the present work 80 specimens were milled all at once under reproducible conditions in every case for each of the four series of measurements.

The presumed reason for the scatter of the experimental results was the effect exerted by atmospheric humidity in the time elapsing between injection moulding and testing. Immediately after demoulding, the mouldings are collected in airtight vessels containing a desiccant. Before testing, the mouldings are taken from the containers and after deflashing they are inserted in magazines, and the notches are milled. The completed specimens are then immediately transferred into other magazines and placed in an automatic test equipment, where the residual width and the impact strength are measured automatically. Even if the greatest care is taken and no time is lost, it cannot be avoided that the specimens' outer fibres, in which the initial crack is formed, absorb different amounts of moisture during all these handling operations.

Since the melt viscosity of nylon 6 and nylon 66 is comparatively low, good prospects of avoiding these handling operations were held out by incorporating the notches during the injection-moulding operation. This was realized by means of a four-cavity mould (Fig. 8) which allowed the notch radius to be set to $r = 1.50 \pm 0.05$ mm. No differences could be discerned in the mechanical properties of the mouldings in each of the four cavities. The sink mark between the two notches lies within the tolerances.

In order to allow a comparison between notches incorporated during injection moulding and notches introduced by milling, 50 mm × 6 mm × 4 mm specimens were injection-moulded without notches from the four products mentioned above. Afterwards, the mould was changed and corresponding specimens with notches were injection-moulded. Notches were subsequently milled in the first set of specimens. Both

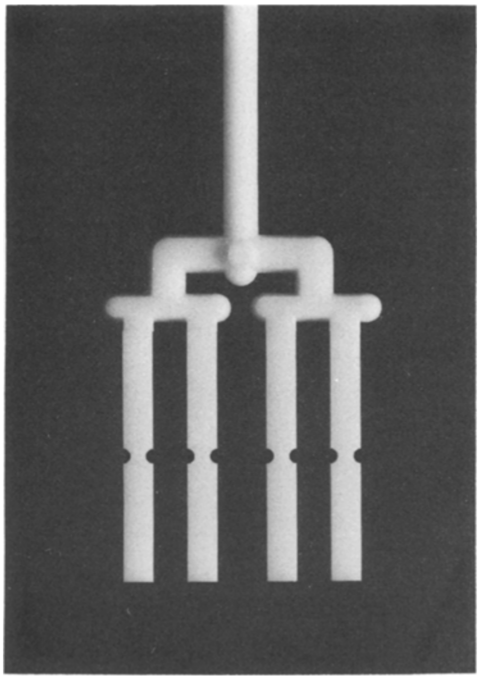

Figure 8 50 mm × 6 mm × 4 mm standard bar specimens with injection-moulded double-V notches ($r = 1.5$ mm) for the DIN 53 753 test.

sets of specimens were subjected to the impact test (notch radius $r = 1.5$ mm) at 60 ± 15 minutes after they had been demoulded. This time schedule is essential to ensure good reproducibility or comparability. When it was strictly adhered to, the figures for the impact strength of nylon 6 specimens with milled notches showed no difference to those with moulded-in notches; some difference was observed in nylon 66 specimens, the values for those with the moulded-in notches being 5 to 9% higher. These findings appeared to be acceptable, because specimens with moulded-in notches offer a great advantage in testing dry polyamides; since the post-moulding manipulations are restricted to deflashing and the dimensional stability of the specimens is ensured, the variation coefficient can be reduced to below 5%. Thus conditions exist for a more accurate study of the repeatability.

4. Repeatability and reproducibility

The first experiments performed to check the repeatability by means of the impact test were discouraging. All the factors that might have influenced the results had to be eliminated step by step. The reason was thus found to be the instability of the freshly injection-moulded ("as-moulded") state. This was determined by keeping a stock of injection-moulded specimens at 23° C in an atmosphere from which moisture was meticulously excluded. The impact strength a_v ($r = 1.5$ mm) was then determined after various periods of time. It was thus discovered that the impact strength decreases exponentially with time as shown in Fig. 9, which was plotted from figures obtained on the particularly sensitive normal-setting nylon 6.

If the initial value of impact strength at, say, 2 to 3 h after demoulding is taken as the reference, it can be seen that the effect is more pronounced in the products that solidify at a normal rate than in nucleated products, and more pronounced in nylon 6 than in nylon 66. This is shown in Fig. 10. Further studies revealed that the effect in each of the four groups investigated decreases with increasing viscosity number. The results can be expressed in another way, namely the greater the impact strength of the freshly injection-moulded specimens, i.e. 1 h after demoulding, the more pronounced is the effect.

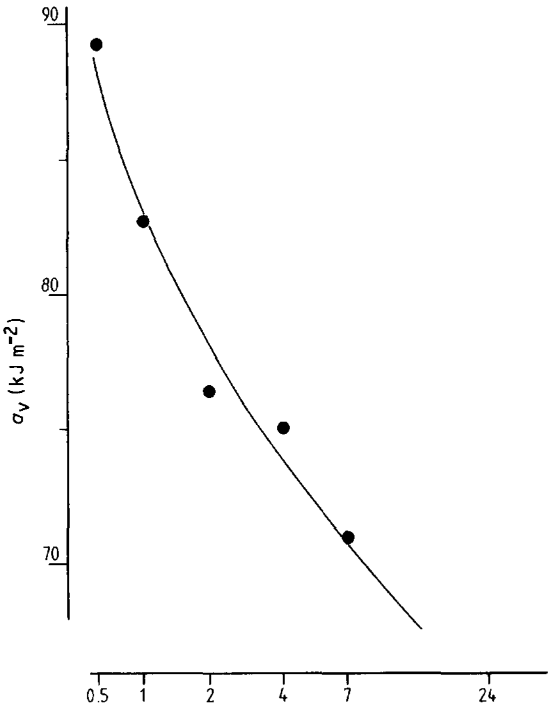

Figure 9 Instability of the freshly injection-moulded state; DIN 53 753 impact strength ($r = 1.5$ mm) as a function of time after demoulding.) The example taken was a normal-setting nylon 6.

Even after 70 days (Fig. 10), the change in impact

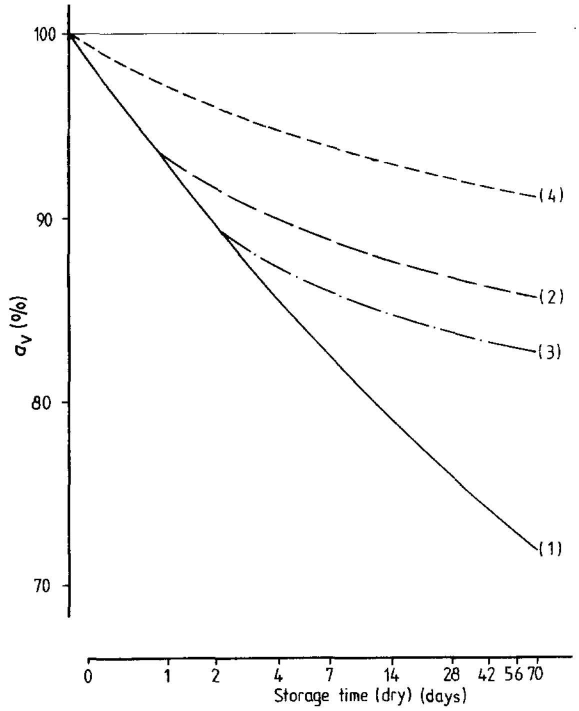

Figure 10 Change in impact strength (a_v, $r = 1.5$ mm) of freshly injection-moulded nylon as a function of the time between demoulding and testing: (1) nylon 6 normal setting, (2) nylon 6 nucleated, (3) nylon 66 normal setting, (4) nylon 66 nucleated.

TABLE II Repeatability of impact strength a_v (r = 1.5 mm, age of specimen 2 to 3 h)

Date specimen prepared	a_v (kJ m^{-2})
14 July	71.4 ± 2.9
16 July	71.0 ± 3.2
28 July	70.0 ± 2.9
11 August	70.4 ± 4.5
12 August	70.5 ± 1.8

strength has not yet been completed. The impact strength for normal-setting nylon 6 that has been stored for three months in a dry atmosphere approaches the figure for nucleated products (with the same viscosity number) in the "as-moulded" state. The change in impact strength is accompanied by an increase in rigidity of specimens kept under dry conditions, i.e. by an increase in the modulus of elasticity in tension and flexure.

Hence the only defined state is that immediately after demoulding. In order to check its reproducibility, dry pellets from the same drum were placed in a number of moisture-proof canisters. Each of these identical samples was injection-moulded in different machines to 50 mm × 6 mm × 4 mm notched specimens, on which the impact strength was measured 2 to 3 h after demoulding. The results listed in Table II represent a typical series obtained with the particularly sensitive normal-setting nylon 6. If the time schedule is strictly adhered to and the injection-moulding machines are clean (see below), the DIN 53 753 impact test with double-V notches (r = 1.5 mm) satisfies the requirements imposed at the beginning on a routine check for the toughness of unreinforced nylon 6 and 66 in the dry state. However, in order to arrive at reproducible test results by this means, the "as-moulded" state must be defined more precisely in future.

5. The "as-moulded" state

Up to now, the most that has been quoted in publications and standards on the state of dry specimens is the statement"as-moulded". No information is given on the age of the specimens at the instant when they were tested.

In view of the results obtained, the recently revised version of the German standard for nylon moulding compounds (DIN 16 773 Teil 2-1984) lays down that freshly injection-moulded specimens be tested after 7 ± 1 days. As is evident from the exponential decrease of impact strength with increasing age of the dry specimens in the repeatability tests described above (Fig. 11), not very much has been gained in specifying this period of time.

It can be seen that the rate of change in the state of the specimens differs on each repetition. One hour after demoulding, no difference can be detected in the figures for the impact strength, but the values measured after seven days are decidedly scattered. One of the probable reasons for these differences in the rate of change in the state of the specimens may be the presence of contaminants that exert a nucleating effect and originate from the injection-moulding machine. Evidence for this is given by the example shown in Fig. 11. In this case, the properties of the moulding compounds were entirely changed by contaminants that were visible to the naked eye and originated from deposits in the injection-moulding machine, which had not been cleaned before the test.

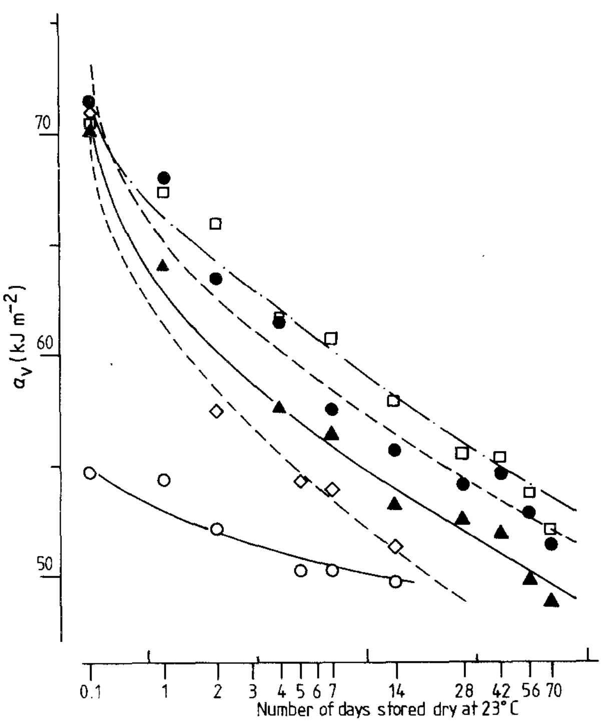

Figure 11 Reproducibility of injection moulding, measurement of impact strength (r = 1.5 mm) and change in the freshly injection-moulded state as a function of the time that the specimens were kept dry at 23° C. The example chosen was normal-setting nylon 6. Symbols refer to different mouldings of the same material; (○) showed the presence of contaminants.

6. Regeneration of the "as-moulded" state

Regardless of whether the term "as-moulded" is limited to a few hours or a few days after demoulding, any specification as to the time when a test should be performed can be realized only by those companies that prepare the injection-moulded specimens themselves. This has raised the question of whether the change in state is reversible and whether the original state can be regenerated.

Systematic studies have demonstrated that the change in state cannot be ascribed to post-crystallization, i.e. irreversible ageing in the sense of DIN 50 035. The more probable reason is compaction of the amorphous phase (increasing density), and it is assumed that this can be reversed above the glass temperature T_g. The assumption has been confirmed in conditioning experiments.

Heating for brief periods to temperatures above the glass transition suffices for regeneration. In order to avoid irreversible changes, the temperature should not be too high. The time required for heating is immaterial, whether it be of the order of a few minutes or a few weeks. What really matters is that the glass transition range should be traversed at a defined slow cooling rate, e.g. 10 min K^{-1}. During cooling, mutual

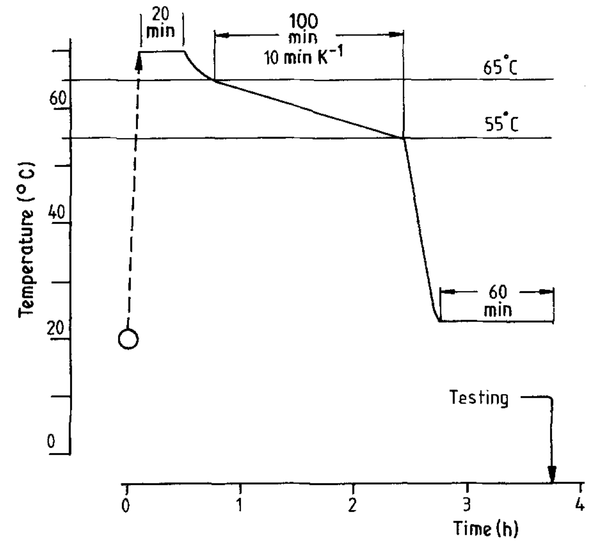

Figure 12 Temperature–time programme for the regeneration of the freshly injection-moulded state of nylon (condition reached about 1 h after demoulding).

shielding of the specimens should be avoided, e.g. by using open magazines. An annealing programme that has proved to be satisfactory is shown in Fig. 12.

If samples are taken from a stock of dry specimens at different times and subjected to the temperature cycle shown in Fig. 12, the impact strength a_v, determined 1 h after they have cooled to 23° C, will always be the same as that of a specimen in the "as-moulded" state (1 h after demoulding) regardless of their age before the beginning of the regeneration process. This is demonstrated in Fig. 13. At higher conditioning temperatures, e.g. 90° C in Fig. 13, the level of impact strength is shifted in the direction of higher values.

It was confirmed that the state of all of the products investigated here could be regenerated in this manner.

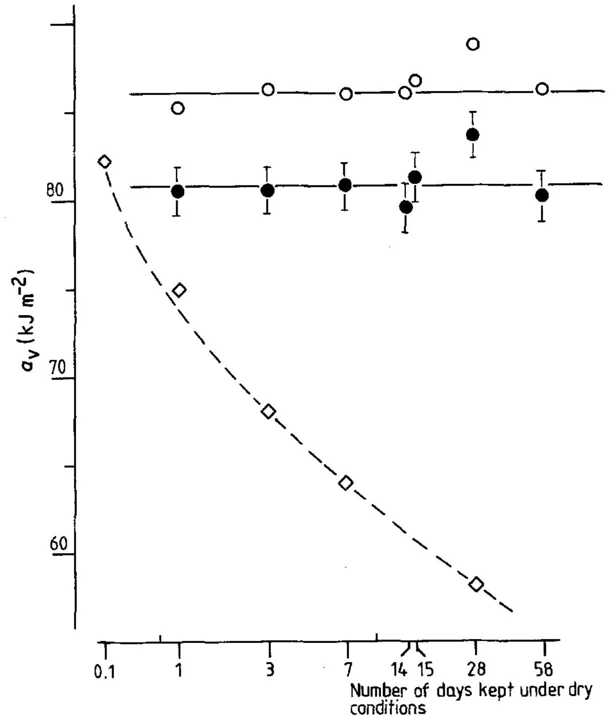

Figure 13 Regeneration of the freshly injection-moulded state: impact strength (r = 1.5 mm) as a function of the age of the specimens. The example taken was normal-setting nylon 6. (○) Annealed at 90° C, (●) annealed at 70° C, (◇) impact strength (dry) before annealing.

Even dry specimens that were 3 years old could be converted into the same state as that which they had 1 h after demoulding. After the test specimens have been annealed in accordance with the programme in Fig. 12, the change in state as illustrated in Figs 10 and 11 commences once again, but the annealing process can be successfully repeated several times afterwards.

Thus annealing offers the opportunity of converting dry specimens of unknown age into a defined "as-moulded" state that can be taken as a basis for comparison. The same applies, in principle, to specimens that are no longer quite dry, i.e. with a moisture content of 0.3 to 0.4%, if they are re-dried in a current of nitrogen at 70° C before annealing.

In contrast to the Charpy test with a U-notch (ISO 179/2C) and Izod test (ISO 180), the impact test with double-V notch according to DIN 53 753 yields a value of impact strength for unreinforced, dry nylon 6 and nylon 66 that can be considered comparable to the impact behaviour in practice. If the test specimens are injection-moulded with double-V notches, the reliability of the measurements will be sufficient for quality control.

But the results of this impact test do not allow comparison unless allowance is made for the instability of the "as-moulded" state of the moulding material. This is best ensured by performing the test 1.0 ± 0.25 h after the specimens have been demoulded. If such tight schedules cannot be adhered to, a defined annealing programme allows dry specimens of unknown age to regain the "as-moulded" state that they had about 1 h after they had been demoulded.

Thus the basic condition is satisfied for investigating whether a definite relationship exists between the impact strength measured on dry specimens and that measured on moist specimens.

7. Reference state of moist nylon

Adherence to a defined "as-moulded" state is an essential basis for comparing the results of tests on dry nylon. The same applies to nylon in the moist state. Absorption of moisture gives rise to morphological changes which greatly change the properties. First of all, the water molecules replace the hydrogen bonds acting between the amide groups of neighbouring segments in a chain. This bonding of the absorbed water is favoured by the energy balance. Once the hydrogen bonds in the amorphous zones of the nylon have been completely loosened, any water that is further absorbed is bonded in only a loose form and acts primarily as a plasticizer.

Hence the toughness and rigidity of nylon 6 and nylon 66 do not change steadily with increase in moisture content. For instance, the modulus of elasticity at 23° C for nylon 6 remains almost unchanged as the moisture content rises to about 2.5%. Afterwards, when further moisture has been absorbed in amounts of only a few tenths of one per cent, it drops to a fraction of the initial value [4]. Thus the irregularity occurs just below the equilibrium moisture content in a standard laboratory atmosphere of 23° C and 50% relative humidity.

Hence, the requirements imposed on a basis upon which tests can be compared are not merely restricted to a given moisture content; the moisture content must also be in equilibrium with the environment. Under given conditions of temperature and water vapour partial pressure, the outer layers of a nylon article become rapidly saturated in the initial stages of water absorption. A comparatively clearly defined boundary is formed between these outer layers and the dry core. As absorption progresses, this boundary gradually moves inwards at a rate of about 0.02 mm per day at 23° C [5]. It is not until the moisture content is in equilibrium throughout the entire cross-section of the specimen that one of the conditions is fulfilled for a comparable moist state. However, as will be demonstrated below, this is still not sufficient.

For practical reasons, the DIN and ISO panels dealing with the standardization of polyamides have agreed that the reference state for moist polyamides should be the equilibrium or saturation moisture content ("conventional" moisture content, see ISO 1874/2) that is attained in a standard laboratory atmosphere of 23° C and 50% relative humidity. The agreement is unrealistic for routine tests because, for instance, a standard bar specimen (cross-section 6 mm × 4 mm) could not attain equilibrium moisture content in a standard laboratory atmosphere even if it were kept there for 360 days. As a compromise, it has been laid down in ISO 1874/2 that specimens subjected to accelerated conditioning according to ISO 1110-1975 can also be regarded as having the conventional equilibrium moisture content.

Thus adjustment of the moisture content to a reference level was tied to the accelerated conditioning procedure as laid down in ISO 1110-1975. When this was checked, however, it was discovered that the method specified in this standard did not lead to a reproducible reference level. Hence, ever since 1977, the pertinent DIN and ISO panels have been occupied in devising another, more suitable method.

8. Procedure for accelerated conditioning

If the water content is not too high, the rate of water absorption obeys the second Fick's law [6, 7]. The only effective means of accelerating the thermodynamic, activated process involved is to raise the temperature. The main methods that could be considered for the necessary revision of ISO 1110-1975 are listed in Table III.

Some of the methods listed in Table III can be ruled out for following reasons:

1. Method A, because the solution of potassium acetate contaminates the specimens and renders them useless mainly for the determination of electric properties.
2. Method B, not only because it requires too much time but also because the moisture content has to be adjusted according to the equilibrium moisture content in a standard atmosphere as determined beforehand on a small splinter of a test specimen. This value, together with a conditioning temperature of 40° C, leads to results that are of no use, as will be demonstrated below.
3. Method C by no means conditions the specimens. All that it achieves is oversaturation of a very thin outer layer; the core remains dry. The state is undefined and not reproducible.

Methods D and E make use of an unsuitable technique. Adjusting a given water vapour partial pressure in an enclosed space, namely a desiccator, above

TABLE III Methods for accelerated conditioning of nylon

Method	Laboratory atmosphere		Duration* (days)	Brief description
	Temperature (° C)	r.h. (%)		
A†	95	?	5	Immersion in potassium acetate solution of a given concentration at 95 to 100° C
B‡	40	92	~80	Exposure to atmosphere of 40° C and 92% r.h. until the specified amount of water has been absorbed. Afterwards, kept for 8 weeks until equalization
C§	50	100	(2)	48 h immersion in water at 50° C followed by 1 h in atmosphere of 25° C and 50% r.h.
D¶	70	62	10 to 20	Kept at 70° C above a saturated solution of potassium iodide
E[a]	50	70	15 to 30	Kept at 50° C above a mixture of glycerol and water of given concentration
F[b]	95	50	2 to 4	Air-conditioned cabinet
G[c]	70	62	10 to 12	Air-conditoned cabinet (ISO/DIS 1110-1985)

*Typical values for nylon 6 of 4 mm thickness.
†ISO 1110-1975.
‡DIN 53 714-0.5.78.
§Ford Engineering Material Specification S-M4D 4584 (1981).
¶Proposed standard submitted by Siemens (1978).
[a] Proposed standard submitted by C.-F. Kuo, Shanghai Plastics Research Institute (1981).
[b] Draft standard NF T 51-051-12.82.
[c] Proposed standard submitted by BASF Aktiengesellschaft (= ISO/DP 1110-1982).

defined solutions (as in DIN 50 008 Teil 1 and 2, ISO/R 483-1966, ISO/DIS 483-1984 and ASTM E 104-51) does not lead to the partial pressures quoted in the literature unless there is no interaction between the object and its environment. However, if dry nylon is placed in an enclosed space of this nature, there is always a concentration and temperature gradient. Hence the time required to achieve equilibrium depends considerably on the spatial arrangement of the specimens, the geometry of the enclosed space, and the degree of fill. Every time the desiccator is opened, e.g. to check the specimens' change in weight, the atmosphere is disturbed and the equilibrium state thus cannot be reliably recognized.

The only conditioning technique that can lead to a defined reference state is placing the specimens in an air-conditioned cabinet with a closed air circuit. It has then to be determined which of the three atmospheres suggested to the standardization panels, namely 50° C/70% r.h., 70° C/62% r.h. or 95° C/50% r.h., gives rise to a state that corresponds to equilibrium moisture content in a standard laboratory atmosphere of 23° C and 50% r.h.

The amount of water absorbed at equilibrium in the air-conditioned atmosphere proved to be an unsuitable criterion. The very unreliability of the data given in the literature on the equilibrium moisture content of nylon in conditioning atmospheres of 23° C and 50% r.h. ([7] and DIN 53 714-05.78) indicates that the state of the nylon before conditioning exerts a certain influence in investigations of this nature. For instance, a difference in state already occurs if the same moulding material is injection-moulded under identical machine settings into two different moulds. As an example, the figures given in Table IV were obtained under these conditions in dry nylon 6 specimens that were a few weeks old.

In addition, the injection-moulded state is unstable, as has been demonstrated above in the light of impact strength measurements.

Now the morphological state of the nylon not only influences the equilibrium moisture content in a standard laboratory atmosphere of 23° C and 50% r.h. but also the rate of diffusion and thus the amount of moisture absorbed in a given time. In order to illustrate this, specimens were taken at various times from a stock that had been stored under dry conditions. Each was conditioned for 14 days in an atmosphere of 50° C and 70% r.h., then transferred into an atomosphere of 23° C and 50% r.h. Their moisture content was determined 1 h and 7 days after the transfer into the second atmosphere. The results are shown in Fig. 14. Under these conditions, the specimens that were 0.1 day old before conditioning absorbed about 4.2% of water. As the age of the specimens before conditioning increases, the moisture content drops to about 2.8% and increases again when the age reaches 28 days. In addition, all the specimens were oversaturated with respect to the standard laboratory atmosphere of 23° C and 50% r.h., because they lose moisture when they are transferred into this atmosphere.

TABLE IV Measurements on dry nylon 6 [8]

	Specimen ISO 179 Type 2 (50 mm × 6 mm × 4 mm)	Dumb-bell specimen
Density (g cm^{-3})	1.1325	1.1289
Ratio of α to γ crystals	4:1	2:1

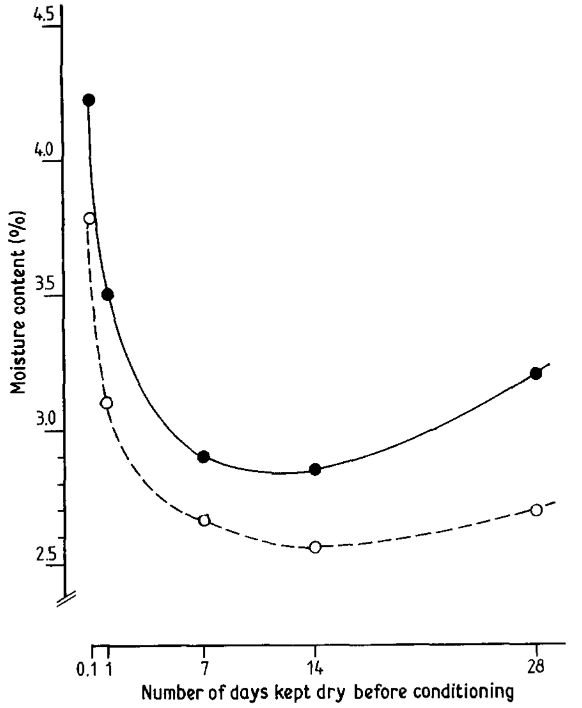

Figure 14 Effect of the specimens' age on the moisture content of nylon 6 (nucleated) after 14 days' conditioning at 50° C and 70% r.h. Measured after (●) 1 h and (○) 7 days at 23° C and 50% r.h.

The values measured for the impact strength of conditioned nylon 6 specimens 1 h, 1 day and 7 days

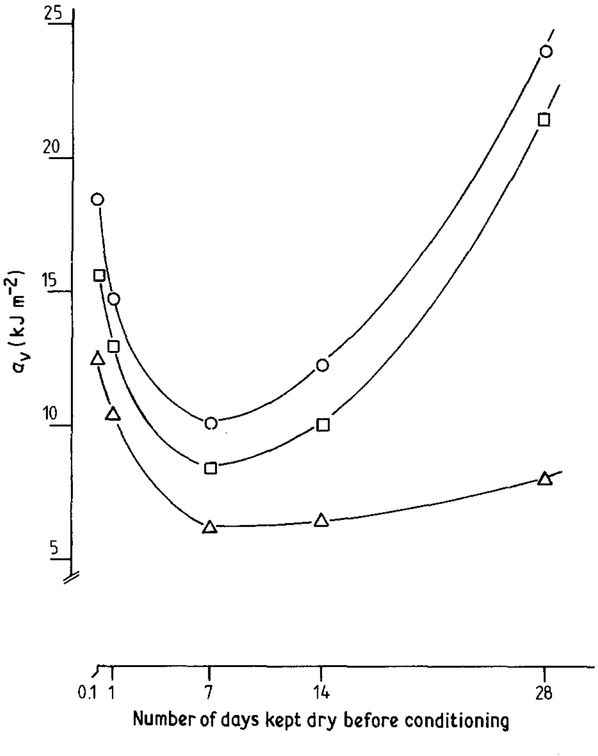

Figure 15 Effect of the age of nylon 6 specimens before conditioning on the impact strength (r = 0.1 mm, ±0° C); tested (○) 1 h, (□) 1 day and (△) 7 days after transfer into an atmosphere of 23° C and 50% r.h. Conditioned at 50° C and 70% r.h. for 14 days.

after they have been transferred into the standard laboratory atmosphere of 23° C and 50% r.h. (Fig. 15) show the effect of the age of the dry specimens on the course of conditioning in an atmosphere of 50° C and 70% r.h. Accordingly, accelerated conditioning that can act as a basis for comparing the properties in the moist state cannot be defined unless the initial state before conditioning is also defined. As has been stated above, the change in the dry state that occurs after injection moulding is reversible at temperatures above the glass transition T_g. Thus comparable conditions for accelerated conditioning could be expected only at temperatures above the glass transition T_g for dry polyamides.

An objection that is continually held against the application of elevated temperatures for conditioning is that they could give rise to recrystallization or post-crystallization, which would preclude a systematic comparison between dry and conditioned specimens. This question was investigated in an ISO round-robin test. The dry specimens for this purpose were about 3 months old. They were ISO 179 Type 2 (50 mm × 6 mm × 4 mm) standard bar and dumb-bell specimens obtained from nylon 6 with a viscosity number of 159 ml g^{-1} and nylon 66 with a viscosity number of 150 ml g^{-1}; they were conditioned in an air-conditioned cabinet with a closed air circuit until their weights were constant under three sets of conditions, namely 50° C and 70% r.h., 70° C and 62% r.h., and 95° C and 50% r.h. (Fig. 16). The following properties were determined on these specimens:

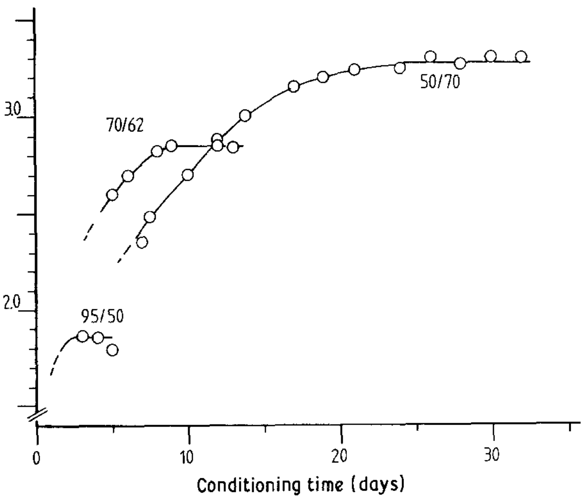

Figure 16 Conditioning curve (change in weight ΔG) for 50 mm × 6 mm × 4 mm nylon 6 specimens in three different atmospheres in air-conditioned cabinets. Figures given (° C)/(% r.h.).

(a) the density in carbon tetrachloride–heptane at 20° C;

(b) the line resolution for the main reflection in the wide-angle X-ray spectrum;

(c) for nylon 6: $\Delta\theta = \theta_{002/202} - \theta_{200}$;

(d) for nylon 66: $\Delta\theta_{010/110} - \theta_{100}$;

TABLE V ISO round-robin test [8]: X-ray studies (for explanation see text)

		Laboratory atmosphere (° C/% r.h.)					
		Nylon 6			Nylon 66		
		70/62	95/50	50/70	70/62	95/50	50/70
50 mm × 6 mm × 4 mm standard bar specimens							
Density	Before	1.1325	1.1325	1.1325	1.1371	1.1371	1.1371
	After	1.1397	1.1396	1.1389	1.1466	1.1449	1.1452
	Increase	0.0072	0.0071	0.0067	0.0095	0.0078	0.0081
Line resolution, $\Delta\theta$	Before	1.55	1.55	1.55	1.50	1.50	1.50
	After	1.66	1.65	1.63	1.68	1.66	1.67
	Increase	0.11	0.10	0.08	0.18	0.16	0.17
Crystallinity:							
W_c	Before	–	–	–	44	44	44
	After	–	–	–	48	43	46
CPI	Before	77	77	77	77	77	77
	After	83	83	82	88	85	85
α/γ (approx.)	Before	4:1	4:1	4:1	–	–	–
	After	5.5:1	5:1	5.5:1	–	–	–
Dumb-bell specimens							
Density	Before	1.1289	1.1289	1.1289	1.1379	1.1379	1.1379
	After	1.1389	1.1375	1.1361	1.1474	1.1459	1.1467
	Increase	0.0100	0.0086	0.0072	0.0095	0.0080	0.0088
Line resolution, $\Delta\theta$	Before	1.54	1.54	1.54	1.53	1.53	1.53
	After	1.61	1.56	1.56	1.71	1.65	1.62
	Increase	0.07	0.02	0.02	0.18	0.12	0.09
Crystallinity:							
W_c	Before	–	–	–	44	44	44
	After	–	–	–	48	46	47
CPI	Before	78	78	78	77	77	77
	After	80	79	78	88	84	82
α/γ (approx.)	Before	2:1	2:1	2:1	–	–	–
	After	4:1	3.5:1	2.5:1	–	–	–

(e) crystallinity W_c from the W_c-V diagram according to Haberkorn *et al.* [9];

(f) crystal perfection index (CPI), calculated from the position of the main reflection according to Dismore *et al.* [10];

(g) the ratio of α crystals to γ crystals (α/γ) in nylon 6.

The results of the study by Haberkorn [8] are listed in Table V.

In all the methods of conditioning, the density increases by 0.007 to 0.010 g cm^{-3}. This cumulative effect can be ascribed, on the one hand, to a slight but definite increase in crystallinity (W_c) and, on the other hand, to an improvement in the crystalline structure (CPI) or to a shift in the α/γ ratio for nylon 6. The main point that concerns the query raised above is that this change is not dependent – as is so often assumed – on the conditioning temperature; on the contrary, the changes at 95° C are less than those at lower temperatures.

Thus there are no objections against conditioning dry nylon 6 and nylon 66 at temperatures above the glass transition range. Moreover, as advantage of conditioning at temperatures as high as this is that the same initial state is obtained at the commencement of conditioning (see Figs 12 and 13). A laboratory atmosphere of 50° C and 70% r.h. does not satisfy this requirement and its vapour partial pressure is obviously too high (see Fig. 16). Since a laboratory atmosphere of 95° C and 50% r.h. can also be disregarded, because the water vapour partial pressure is much too low (Fig. 16), the further course of the study was concentrated in a laboratory atmosphere of 70° C and 62% r.h., which came very close towards meeting the demands.

As was stated above, the moisture content cannot be regarded as a criterion for complete conditioning. Thus only the one means remains for checking whether accelerated conditioning has attained a state that corresponds to saturation or equilibrium in a standard laboratory atmosphere of 23° C and 50% r.h. This is that the specimens must not undergo any change in weight, even over longer periods, if they are kept at 23° C and 50% r.h. after conditioning. The extent to which this requirement is met by a laboratory atmosphere of 70° C and 62% r.h. for nylon 6 and nylon 66 is close enough for the purpose of further studies. For instance, 50 mm × 6 mm × 4 mm standard bar specimens of nylon 6 that were conditioned in this laboratory atmosphere underwent a change in weight of only -0.035% after they had been kept for 83 days in a laboratory atmosphere of 23° C and 50% r.h.; the corresponding figure measured for nylon 66 was -0.04%. In the light of these results, accelerated conditioning in a laboratory atmosphere of 70° C and 62% r.h. was proposed as a draft standard ISO/DP 1110-1982, despite the fact that it still does not meet the demands imposed on a reference state, as will be demonstrated below.

9. Repeatability and initial state

In the course of this study, the relationship was continually checked between accelerated conditioning in a laboratory atmosphere of 70° C and 62% r.h. and the age of the specimen before conditioning. At first, no explanation could be offered for the outliers that occurred.

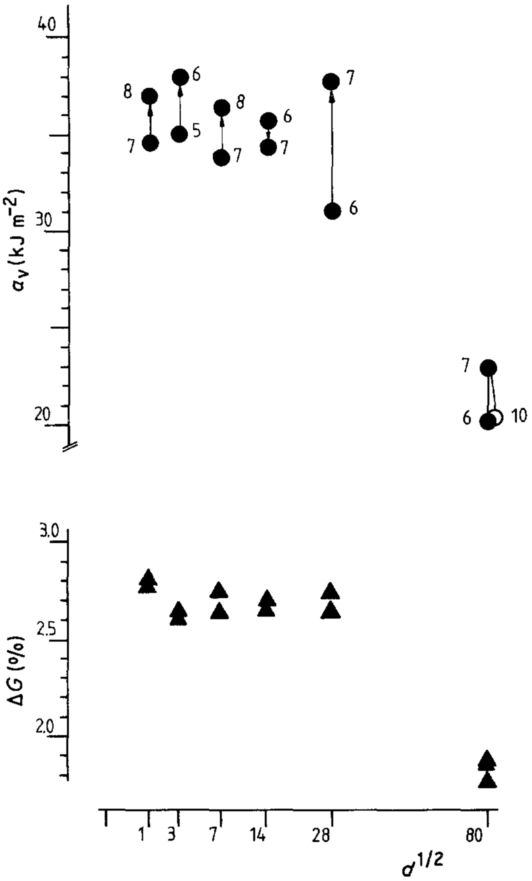

Figure 17 Reproducibility of conditioning at 70° C and 62% r.h. as a function of *d*, the number of days kept dry before conditioning. The example taken was nylon 6 nucleated. Impact strength (r = 0.1 mm, 23° C) measured 1 h after transfer to 23° C and 50% r.h. Figures give days at 70° C and 62% r.h.

In these series of experiments, a stock of specimens were notched immediately after injection moulding and then kept dry in a canister. A series that has been taken as an example is shown in Fig. 17. After the periods of time plotted on the horizontal axis, specimens were withdrawn from the canisters and conditioned until the equilibrium state was attained (see below). One hour after adaptation to an atmosphere of 23° C and 50% r.h., the increase in weight, expressed in terms of the initial weight, and the impact strength were determined. In this example, the reproducibility was good for specimens as old as 28 days. As opposed to this, specimens with an age of 80 days absorbed only 1.8% instead of 2.7% of moisture even after they had been subjected to accelerated conditioning for 10 days, and the impact strength a_v attained a value of only 22 kJ m^{-2}. It transpired that the reason for this difference in behaviour was an irregular increase in the moisture content during the "dry storage" period.

If the specimens are kept in canisters above silica gel, some fresh air is always admitted whenever one is withdrawn. Once the canister has been reclosed, the silica gel rapidly absorbs the moisture that has thus been introduced. However, the water vapour partial

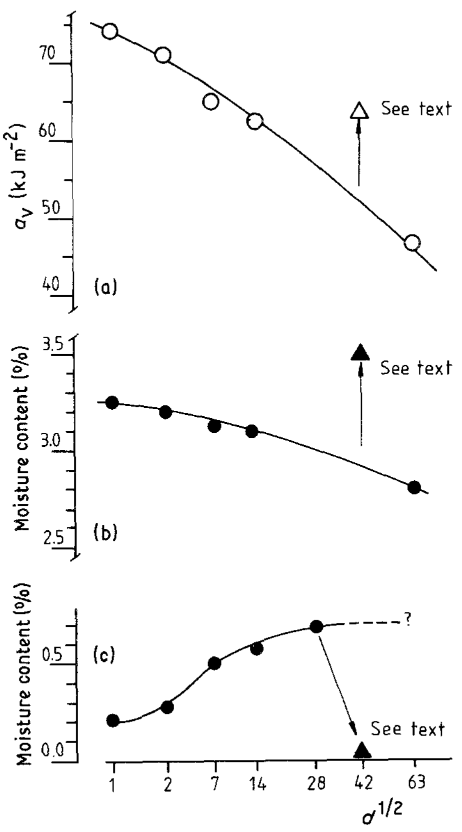

Figure 18 Effect of initial moisture content before conditioning on the results of conditioning for 7 days at 70° C and 62% r.h. The example taken was normal-setting nylon 6. (a) Impact strength (r = 0.1 mm, 23° C) measured 1 h after transfer to 23° C and 50% r.h., (b) moisture content *after* conditioning, (c) moisture content before conditioning. d is the number of days kept "dry" before conditioning.

pressure above silica gel is higher than that above dry nylon 6 and nylon 66. Consequently, if the canister is repeatedly opened and closed, the moisture content of the specimens will increase to a measurable extent. The results of a typical series of experiments that were repeated a number of times are shown in Fig. 18. It can be seen from this that an increase in the initial moisture content before conditioning leads to a decrease in the equilibrium moisture content in an atmosphere of 70° C and 62% r.h. and to an associated decrease in the impact strength as measured 1 h after the termination of conditioning.

If specimens are taken that have become moist (in this case, with 0.65% moisture after 28 days) and are subsequently dried for 14 days in a current of nitrogen at 70° C before they are conditioned, the amount of moisture that they absorb during the subsequent conditioning is even more than the original content. The impact strength also increases but not to the value expected. This problem will be dealt with in more detail later.

Surprisingly enough, it was only moisture that had been absorbed slowly from the air that exerted this effect on accelerated conditioning in an atmosphere of 70° C and 62% r.h. Thus the moisture content of specimens that had absorbed 1.5% of moisture in a normal atmosphere increased by only 0.3% on accelerated conditioning within 14 days. If, however, the dry specimens are immersed in water before conditioning, they release the excess moisture very rapidly in an atmosphere of 70° C and 62% r.h., and the equilibrium moisture content is attained in the normal time. Until an explanation has been found for this paradox, a condition that must be specified for reproducible accelerated conditioning is that the specimen must be kept dry until conditioning commences as in ISO/DIS 1110-1985.

10. Reference state

All that has been dealt with above has been the repeated attainment of equilibrium moisture content. At the same time, the criterion for the quality of the equilibrium state achieved by accelerated conditioning was the impact strength as measured according to DIN 53 753. In the conditioned state, the relaxation time is reduced to such an extent at 23° C that double-V notches with a radius of r = 0.10 mm are required to ensure definite failure of nylon 6 and nylon 66 with a conventional moisture content. It has thus been demonstrated that a moisture content corresponding to equilibrium in a standard laboratory atmosphere of 23° C and 50% r.h. is not, by itself, a valid criterion for a reference state that can be considered as a suitable basis for the comparison of various properties.

The first step to be taken in ensuring accuracy is to lay down tolerances for the equilibrium condition. A maximum periodic and spatial temperature difference of ±1 K has been laid down for the conditioning cabinets with closed air circuits that are on the market. The tolerance permitted by ISO/DIS 1110-1985 for the psychrometric temperature is ±0.3 K.

Equilibrium is checked by weighing out to within an accuracy of ±0.1 mg at least two specimens from each sample to be conditioned and by placing these specimens in different locations in the space available. After the specimens have been conditioned for about 6 days, their weight is checked for the first time 1 h after adaptation to a standard laboratory atmosphere of 23° C and 50% r.h. These checks are repeated at intervals of 1 to 2 days. When the difference in four consecutive checks is less than ±0.05% of the mean value of these four weighings, equilibrium moisture content is considered to have been attained (ISO/DIS 1110-1985). It is advisable to observe the equilibrium for a somewhat longer period still and to take into consideration that the amount of moisture absorbed varies somewhat within a sample and is not quite independent of the position of a specimen in the available space. This even applies if the specimens are kept upright and isolated from one another, e.g. by clamping them in the rails only on one side.

It can be seen from Fig. 19 that the specimens' state has not yet stabilized after the equilibrium moisture content has been reached. In this example, the gain in weight after conditioning for 7 days was 2.83%; thus, since the initial moisture content was 0.18%, the final moisture content was 3.01%. This clearly represents saturation in an atmosphere of 70° C and 62% r.h. However, the impact strength a_v (r = 0.1 mm) measured after equalization for 1 h in a standard laboratory atmosphere of 23° C anmd 50% r.h. is still

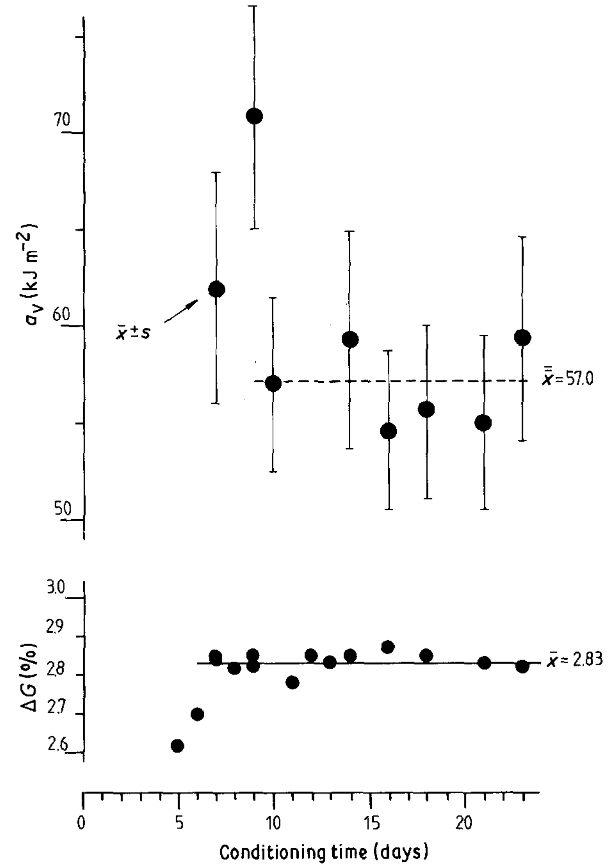

Figure 19 Moisture content and impact strength (r = 0.1 mm, 23° C) of normal-setting nylon 6 as a function of the conditioning time at 70° C and 62% r.h. $\bar{x}$ is the average of a group of measurements, s their standard deviation and $\bar{\bar{x}}$ the overall average of a series.

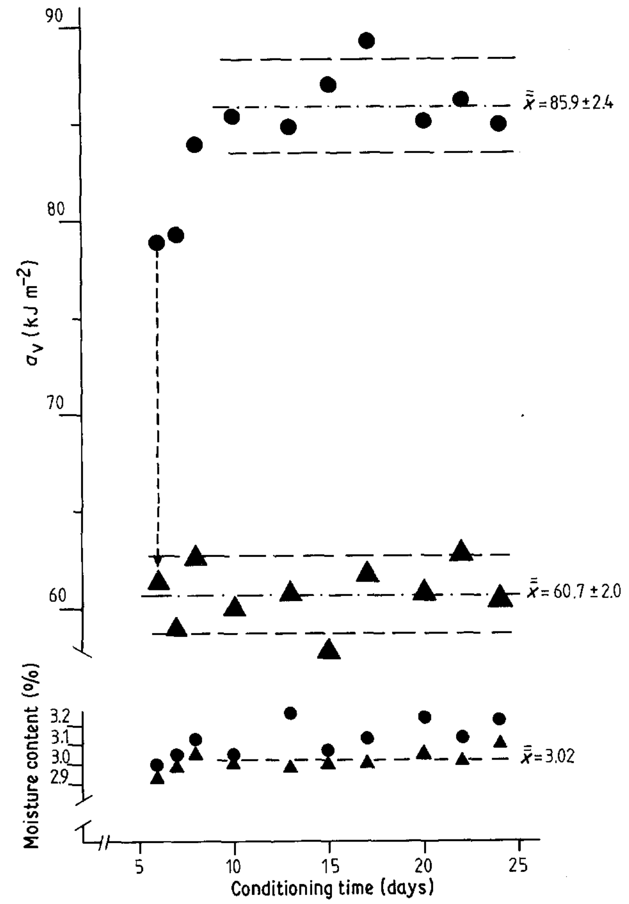

Figure 20 Instability of the state after accelerated conditioning at 70° C and 62% r.h. Impact strength (r = 0.1 mm) measured at 23° C. The example taken was nylon 6. Measurements after (●) 1 h and (▲) 56 days at 23° C and 50% r.h.

unreliable at first. The mean values skip over a wide range with variation coefficients of 15 to 20%. The sensitive criterion selected here, namely the impact strength a_v measured after 1 h at 23° C and 50% r.h., does not become stable unless conditioning is continued for at least 3 days after the saturation point has been reached. It is not until then that the variation coefficient of the mean value is reduced to 8%. It appears that this time is necessary in order to relieve the stresses caused by moisture absorption [11] and morphological changes.

Thus a state can be achieved by conditioning specimens in an atmosphere of 70° C and 62% r.h. until equilibrium moisture content has been attained for at least 3 days longer, and by following up with an equalization period of 1 h in a standard laboratory atmosphere of 23° C and 50% r.h. This state could be taken as the reference for nylon with "conventional" moisture content, because in a series of tests, such as those represented in Fig. 19, the change in weight undergone by the specimen is insignificant, even after months in a standard laboratory atmosphere of 23° C and 50% r.h.

Unfortunately, this state still cannot be satisfactorily reproduced. For instance, rather too much moisture may sometimes be absorbed in the initial stages of conditioning at 70° C and 62% r.h. and be released again as conditioning proceeds. If the specimens are withdrawn too soon from this atmosphere, the moisture content will drop within a few weeks when they are placed in a standard laboratory atmosphere of 23° C and 50% r.h., e.g. from 3.1 to 2.9% (for normal-setting nylon 6). It has not yet been clarified whether the age or the morphological condition of the specimens is responsible.

There are signs to the effect that the tolerances mentioned for the 70° C/62% r.h. atmosphere, namely a psychrometric temperature difference of 10 ± 0.3 K, are too wide. Within these tolerances, the amount of moisture absorbed might be higher than the equilibrium content at 23° C and 50% r.h. (Fig. 20). However, the important point is that the state achieved 1 h after conditioning is still unstable, even if the equilibrium moisture content attained by accelerated conditioning at 70° C and 62% r.h. corresponds to the equilibrium at 23° C and 50% r.h. Even if the moisture content does not change in the atmosphere at 23° C and 50% r.h., a change in state does occur that leads to a definite decrease in impact strength within a few weeks. Evidently, the transfer from 70° C to 23° C initiates fresh morphological changes, although the moisture content remains the same. An example of the consequences is a systematic reduction in the elongation at break as determined in the impact test.

This can be illustratged by conditioning a large number of specimens in an atmosphere of 70° C and 62% r.h. and transferring samples of the specimens from this atmosphere to an atmosphere of 23° C and

50% r.h. at intervals of a few days. From each lot one sample is subjected to the impact test 1 h after the transfer; and the others, after they have been kept for several weeks at 23° C and 50% r.h. The impact strength for the samples tested 1 h after the transfer reaches a certain level after about 10 days conditioning time, as shown in Fig. 19. This level remains fairly constant even after conditioning for 28 days. As opposed to this, the impact strength of conditioned specimens tested after they had been kept longer at 23° C and 50% r.h. dropped to a level that was about 30% lower in the course of a few weeks.

The entire problem is summarized in the one example shown in Fig. 20. The plot for the specimens that were tested 1 h after the transfer to 23° C and 50% r.h. attains a certain level of impact strength after about 10 days of conditioning at 70° C and 62% r.h. analogous to the case shown in Fig. 19, but the level attained is substantially higher. This level dropped from 86 to 61 $\mathrm{kJ\,m^{-2}}$ within a period of 8 weeks after the transfer to the atmosphere of 23° C and 50% r.h. Within the same period, the moisture content evens out at 3.02%.

Within the tolerances quoted for the atmosphere of 70° C and 62% r.h. and under reproducible conditions, decidedly different levels exist for the impact strength as measured 1 h after conditioning. Within a period of 2 to 8 weeks at 23° C and 50% r.h., the initial level of impact strength always drops by about 30%, even if the moisture content is exactly equivalent to the equilibrium value in this atmosphere. As was the case with the specimens in the dry state, as described above, the rate at which the state changes is not reproducible. When the specimens have been kept 2 to 8 weeks at 23° C and 50% relative humidity after accelerated conditioning, the level of impact strength is of the same order of magnitude as that which would be reached in about 400 days at 23° C and 50% r.h. without accelerated conditioning.

Hence, at the present stage in the study, a reference state cannot be attained by accelerated conditioning with the method laid down in ISO/DIS 1110-1985 and that would act as a suitable basis for a comparable determination of the toughness of specimens with "conventional" moisture contents. The reproducibility requirements imposed on the state attained 1 h after conditioning are still unclear. In addition, this state is unstable. At the present time, it is being investigated whether it is possible to reproduce the state that is attained after accelerated conditioning followed by storage for a few weeks in a standard laboratory atmosphere of 23° C and 50% r.h. In any event, the time required for attaining this state, namely 7 to 9 weeks, would be too long for routine tests.

It must remain an open question what the relationship is between the toughness measured in the dry state and that measured at "conventional" moisture content. The study has revealed that the figures conventionally quoted from the properties of "moist" nylon 6 and nylon 66 merely represent orders of magnitude and are too unreliable for comparing different materials. The elements of doubt that have been demonstrated here ought also to affect the quality control tests adopted for moist or accelerated-conditioned articles.

11. Summary

The Charpy impact test according to DIN 53 753 on specimens with double-V notches and a notch radius of $r = 1.5$ mm is suitable for classifying the toughness of dry, unreinforced nylon 6 and nylon 66 moulding materials. The introduction of specimens with injection moulded double-V notches has improved the accuracy of this test method to the extent necessary for quality control. In the light of this criterion, it has been demonstrated that the "as-moulded" state is unstable and that mainly the toughness and rigidity change in the course of some months. The rate of change is variable, with the result that freshly injection-moulded specimens are in a comparable state for only a few hours. The state in specimens of unknown age can be regenerated by an annealing programme consisting of heating for a short period to 70° C and slowly cooling under defined conditions in the glass transition range.

The reference state laid down in standards for moist nylon is the equilibrium moisture content reached in a standard laboratory atmosphere of 23° C and 50% r.h. ("conventional" moisture content). It is intended to compile a standard for an accelerated conditioning method that will allow this state to be achieved for routine measurements. Since the extent and rate of water absorption depend decisively on the change in state of freshly moulded nylon, no consideration need be given to accelerated conditioning methods at temperatures below the glass transition range for (dry) nylon 6 and nylon 66.

It has been proposed that accelerated conditioning in an atmosphere of 70° C and 62% r.h. in a circulated-air oven be adopted as the standard, because the equilibrium moisture content attained under these conditions is very close to that reached in a standard laboratory atmosphere of 23° C and 50% r.h. However, it has been revealed by the DIN 53 753 impact test on specimens with double-V notches (notch radius of $r = 0.10$ mm) that the state attained in an atmosphere of 70° C and 62% r.h. is also unstable and that the toughness and rigidity change significantly over the course of some weeks after the specimens have been transferred from this atmosphere into a standard laboratory atmosphere of 23° C and 50% r.h. It is only after this change has taken place that the properties are comparable with the properties after conditioning in a standard atmosphere of 23° C and 50% r.h. In view of the time involved, the reference state recommended for routine measurements is that attained 1 h after conditioning at 70° C and 62% r.h., but up to now it has not been possible to make this state reproducible.

The study is of considerable significance for the specification of moulding materials and for quality-control tests of finished articles in the dry and conditioned state. It contributes towards a better understanding of the behaviour of nylon parts that are subjected to dynamic loads in practice.

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