

DuPont™ Engineering Polymers

Failure Analysis using Microscopic Techniques



Edith Böhme



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The miracles of science™

Caption for front page illustration:

Thin polished section (10 μm) of a ball pen tip moulded of DELRIN[®] 500 NC010 (Acetalhomopolymer).
Microscopic photography taken in transmittant polarized light using first order red quartz filter.

Magnification: 40 \times

Edith Böhme

Failure Analysis
on Moulded Parts of
DELRIN® and ZYTEL®
using Microscopic Techniques

Note about the author

Mrs. E. Böhme is a Technical Consultant with Engineering Plastic Materials of Du Pont de Nemours International S.A. Geneva.

For more than 20 years Mrs. Böhme has worked with processors and end-users of engineering plastics. During this time she has developed techniques of failure analysis using microscopic structural analysis and demonstrated on countless occasions the value of these studies in understanding part failure.

This work is intended to provide users of engineering materials with a guide to the practical application of this technology.

Introduction

The increasing use of failure analysis on thin sections prepared from finished parts by many laboratories demonstrates that the study of the structure of crystalline material is a useful tool to reduce rejects, optimize moulding conditions and improve the quality of the moulded components. However, once a fairly good thin section has been prepared, it is not always easy to interpret what is seen in the structure and to pinpoint the reason of failure. Experience is needed to be able to identify what has gone wrong in the production, tooling or even designing.

Over the years many cases have come across the authors table and since there are few publications on failure analysis by transmit-tant light microscopy it was the intention to issue a kind of “reference book” to assist the newcomer in this field of technology.

The emphasis is on the pictures rather than on the comments which have been kept short to provide more space for the microscope photos of the thin sections. Some of the failures shown may occur single or in combination within the same part.

The cases described herein demonstrate the usefulness of failure analysis on crystalline materials using microscopic techniques. However, these techniques should not be regarded as *the only* tool for quality control. A thin section of 5-20 μm prepared from a moulded component does not permit a statement about the quality of a whole series. However, if parts fail in testing or in use, an analysis of the structure of a thin section from such a part in comparison to a good one will be of great help in recognizing the possible cause of failure and hence preventing them in the future.

The author realizes that the booklet is only the first step towards a more complete guide book and revisions may be necessary as the techniques advance and more knowledge is gained about the morphology of crystalline materials. We hope and wish that the present version may become useful and handy for day-to-day problem solving.

Acknowledgment

Many moulders and end-users have generously provided us with samples, parts and information of their products and so permitted us to collect a variety of examples. We extend to them all our sincere thanks for their cooperation.

Many thanks to my colleagues who assisted with their efforts and advice towards the accomplishment of this booklet.

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Methods of Obtaining Thin Sections of Semi-Crystalline Polymers for Structural Analysis

Structural analysis using optical microscopy is a method for analysing the reasons for failure of unreinforced semi-crystalline polymers, e. g. polyacetal (POM) and polyamide (PA). It is a technique often used in the laboratories of moulders as well as end-users in the plastics industry.

From systematic studies of the influence of the processing parameters on the crystalline structure of moulded thermoplastic resins in relation to their mechanical properties, data are now available which allow many causes of failure to be determined by structural analysis alone. Several authors in this field³⁻¹⁰ give interpretations of observed phenomena in the structure of moulded parts.

Unfortunately, this failure analysis technique is not used as frequently as it should be because at first it seems to be difficult to prepare a thin and thus light transmittant sample. Failures arising from incorrect processing become visible only in a light transmittant sample with the aid of polarisation filters and sometimes with the additional use of a first order red quartz wave plate inserted in the optical path to further delineate structural differences.

In the following text, two preparation methods are described for obtaining thin, light transmittant specimens. One is the method of cutting a thin section with a microtome, as used in the medical field. The other is a polishing method, a technique with instruments used mainly in metallurgy. A third method, originating from the field of medicine, but also used in metallurgy, is that of micromilling. The use of such a device has been described in publications^{b, c, d, h} and will not be discussed here.

The decision as to which method to use for the preparation of a thin section depends mainly upon the complexity of the object to be analysed and/or the time available for the analysis.

The microtoming technique provides a thin section for microscopic inspection within 10-20 minutes. However, the microtome holding device sets certain limits on the size and complexity of the part to be cut.

The polishing technique provides a thin section for microscopic inspection, but takes a few hours to achieve depending on the choice of embedding resin and the time required to cement the sample on to the glass support. However, the polishing technique allows fractured samples to be observed as a unit and/or at angles which would not have been possible with the sample holder device of the microtome.

The polishing technique usually provides sections having better definition than those obtained by microtoming (see photos for comparison, p. 6).

The preparation of a light transmittant section by the microtoming technique is done in two steps:

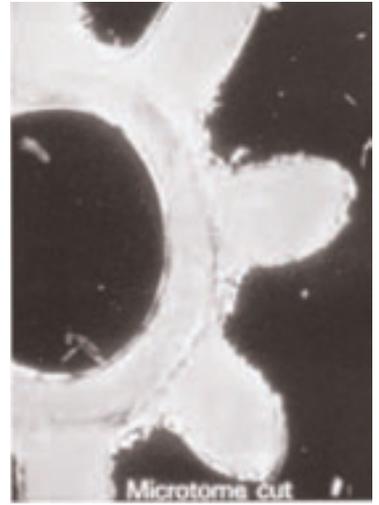
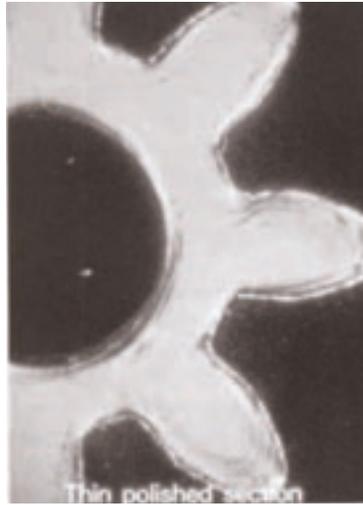
- Cutting with a microtome
- Mounting of the thin section on a glass support for microscopy

Advantages and Disadvantages of Microtome Cutting and Polishing Techniques

A. Cutting Technique (Microtome Cut)

a
Thin Polished Section

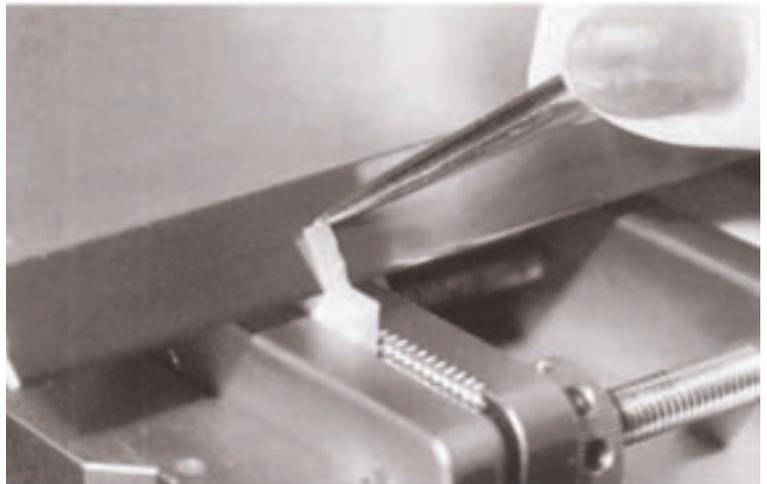
b
Microtome Cut
(done with an already abused
knife)



a

b

*Thin section
cut with the microtome*



Cutting with a microtome

Thin sections can be prepared with all microtomes available on the market provided they have a good solid base. Special attention has to be paid to the cutting knives and the cutting angle of the knife. The knife should be positioned at a 40-45° angle to the part. Tungsten carbide tips are recommended for cutting POM and PA. The part to be sectioned must be mounted rigidly in the sample holder of the microtome, but without applying too much pressure so as to avoid irregular thicknesses within the cut-off section. The free portion of the sample should not exceed 2 mm. The preferred thickness of the section is between 10-20 μm . In some cases *too thin* a cut does not provide the desired information. It is advisable to prepare sections from 25 μm downwards to see in which thickness the failure can be identified best.

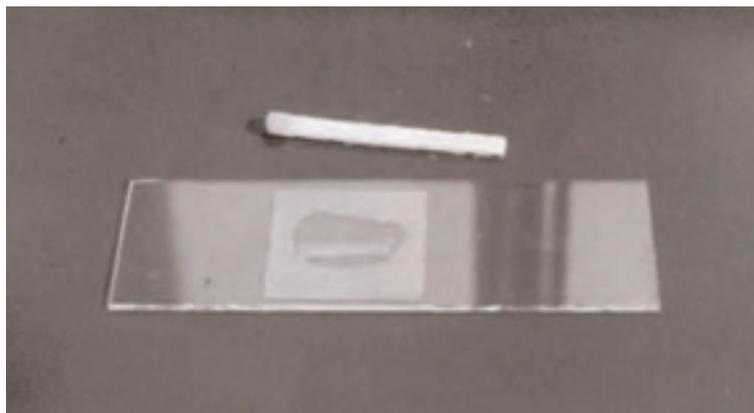
If it is not possible to clamp the part or a section of the part directly in the holder, it may be embedded in a suitable resin and then cut after the resin has hardened. Often the thin section will separate from the embedding resin when cut and can then be mounted on the glass support. If not, the section can be mounted together with the surrounding embedding resin. Another possibility is to embed only part of the sample to be cut so as to provide a solid "foot" to be clamped. For that purpose there are embedding resins on the market which harden quickly within 2-10 minutes. However, the free portion should not be too high in order to prevent bending while cutting.

Mounting on the glass support

Using tweezers, the section is mounted on a microscope slide (76 × 26 mm) in Canada Balsam (neutral in xylene). A drop of the balsam is spread on the slide. The sample is carefully flattened out (a long or thick section will curl severely) and, still using tweezers, is pressed into the balsam. The thinner the cut the less the section has the tendency to curl. Balsam is then spread on the cover glass which is placed over the specimen. The cover glass is pressed carefully either by hand or with tweezers to distribute the balsam evenly.

The slide and coverglass assembly with the thin section in between, positioned horizontally, is heated on a hot plate (50-60°C) for 5-10 minutes. A weight with a flat bottom, cut to the diameter of the coverglass (20 mm Ø and approximately 350 g), is placed on the assembly.

Thin section prepared in Canada balsam



This squeezes out the air bubbles in the Canada balsam. When cooling, the heated weight is exchanged for a cold one of approximately the same dimensions. This quick cooling prevents a retraction of the balsam and a possible recurling of the thin section. As soon as the mounted microtome is cooled it is ready for microscopic inspection.

B. Polishing Technique ^{c, f, h}

The preparation of a thin light transmittant section by the polishing technique is done in four steps:

- Embedding
- First polishing
- Cementing
- Second polishing

Embedding

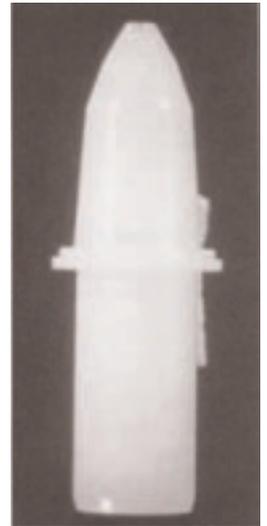
The embedding is done using the procedure established in metallurgy. The choice of embedding resin (Epoxy or Polyester resin) depends on the type of polymer to be encapsulated. Some of the embedding resins develop heat during the hardening process which may effect some of the polymers. With regard to DELRIN[®] and ZYTEL[®] there are no restrictions concerning the choice of embedding resin. Good results have been obtained with EPOFIX (manufactured by Struers, Denmark) and SCANDIPLAST 9101 (manufactured by Scan DIA, Germany). Other products on the market are certainly suitable as well.

*Figure 1:
Textile machine thread guide
in PA 66*

*Figure 2:
Ballpoint pen tip in POM*



1



2

Embedding resins having very little shrinkage are recommended in order to prevent the embedded part falling off when polishing. Some resins harden in a short time with or without exposure to elevated temperatures. Others cure at room temperature after 16 hours or more. To be able to make a failure analysis quickly a resin having a short curing time is preferred.

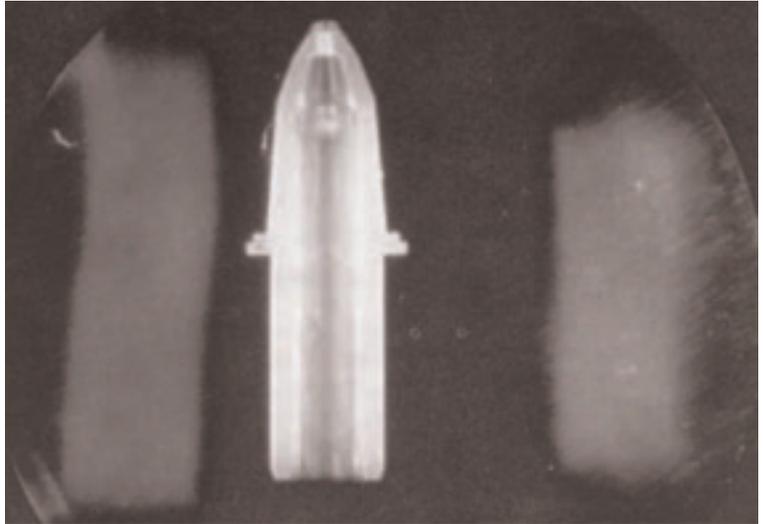
If the part to be embedded contains hollow sections, it is suggested that these are filled with the aid of a disposable hypodermic injection needle before the part is embedded. This procedure prevents the formation of air bubbles in and around the specimen as

well as a possible delamination of the specimen from the glass support during polishing. Should access to the hollow section be difficult, then a vacuum may have to be applied. Small parts can be fixed to the bottom of the container for encapsulation by using a double faced adhesive band to prevent the specimen floating to the surface.

First polishing

The resin with the encapsulated specimen is polished by hand on a wet paper grinder. Starting with a paper SIC-grain of No. 320, the polishing is continued until the level of the desired microscopic observation is reached. Then this surface is ground with increasingly finer grain paper (e. g. 500, 800, 1000, 1200) to improve the quality of the surface for microscopic inspection. If available, automatic wet paper grinding machines are preferred because the overall flatness of the specimen surface will be improved. The better the quality of the specimen's surface the better the adhesion to the glass support in the cementing step. Consequently a better quality of the thin polished sample can be obtained in the second polishing step.

*Ballpoint pen tip
after first polishing*

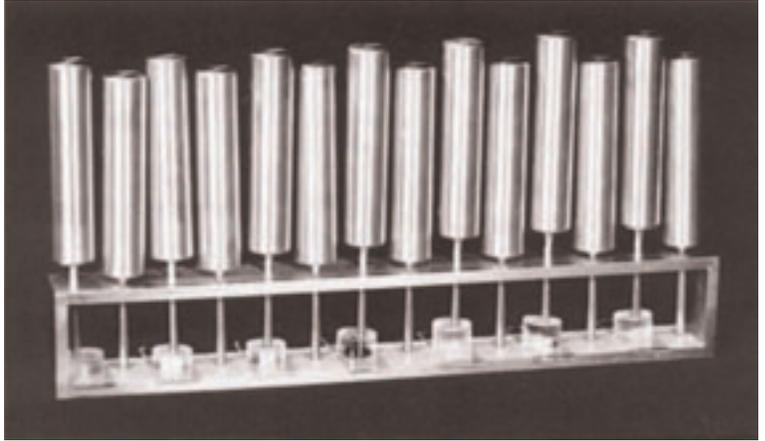


Cementing

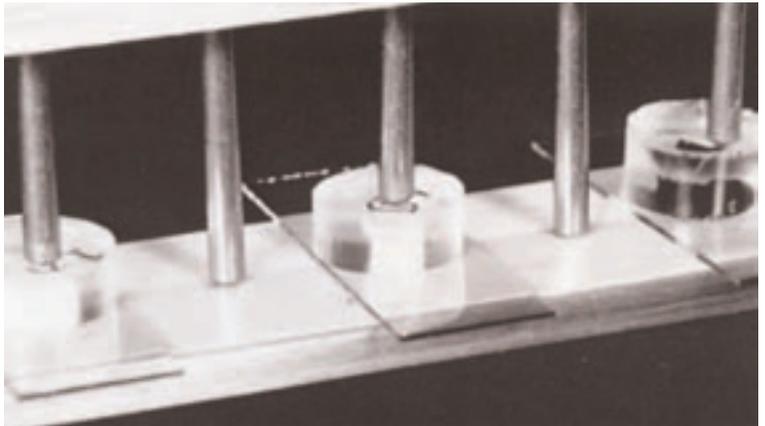
The polished surface of the specimen is now glued on to the glass support (76 × 20 mm). Araldit D with hardener HY956 (manufactured by CIBA-GEIGY) has been used with success for POM and PA. Other glues available on the market may also work well.

The suitability of the cement for each case should be checked beforehand. To achieve a uniform contact between the glass support and the specimen, and at the same time to obtain a uniform thin layer of the cement, a weight of about 500 g is placed on top of the embedded block. Preferably the weight should have a pin in its centre, approximately 5 mm \varnothing and a few centimetres in length, in

Mounting Bloc



Mounting Bloc – Detail



order to apply the load centrally and uniformly onto the specimen (see photos above).

The hardening process can be varied with time and temperature depending on the recommendation of the manufacturer of the cement and the material of the embedded plastic component.

Second polishing

To reduce the polishing time for the thin section, the larger portion of the embedded specimen is cut off with a small saw by cutting just alongside the glass support. The remaining sample of about 1 mm thickness can now be polished as in the first polishing step starting with paper grain size No. 320 and finishing with No. 1200.

The glass support can be held with the aid of a rubber suction cap as used in many garages to polish automotive valves. With this accessory, thin sections of 10 μm thickness and less can be obtained. About 10 μm additional thickness must be accounted for the cement. While polishing, intermediate controls of thickness as well as of the microscopic quality are recommended. As with the microtome

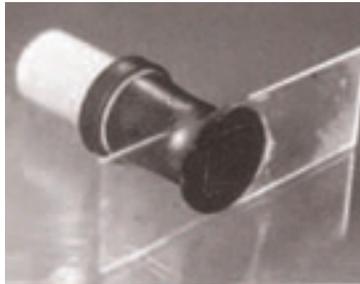
*Figure 3:
Gear (3 mm Ø) after second
polishing step with paper
grain 320*



preparation, sometimes too thin sections may not provide the desired information.

Microscopic inspection of a polished thin section is improved if a small drop of immersion oil is placed between the sample and the cover glass. Should it be necessary to store the thin section for a longer period of time, the oil can be removed with alcohol and Canada balsam is then used in the same manner as for microtomed sections.

*Second polishing with the aid
of a rubber suction cap*



*Figure 4:
Thin polished section of
the textile machine thread guide
(Fig. 1), photographed with
polarized transmittant light*



*Figure 5:
Thin polished section of
the gear (Fig. 3), photographed
with polarized transmittant light*



4

5

Summary

The techniques described are, in reality, simpler to apply than it may seem at first glance. Once they are used and some skill has been achieved, a structural analysis can provide useful additional information for failure analysis studies. For example, when a sudden increase in the number of rejects is noticed in production or in the quality control of delivered parts.

Unmelt or Inhomogeneous Melt

In some cases when moulded parts show unusually low performance or extruded material is brittle when machining, the reason may be the presence of unmolten particles or a non-uniform structure. Unmolten particles might be visible already with the naked eye while the latter will become visible in a thin section inspected with transmittant polarized light.

The reason for the presence of unmolten particles in most cases is a too low or wrong temperature profile for the melt in the barrel of the moulding machine.

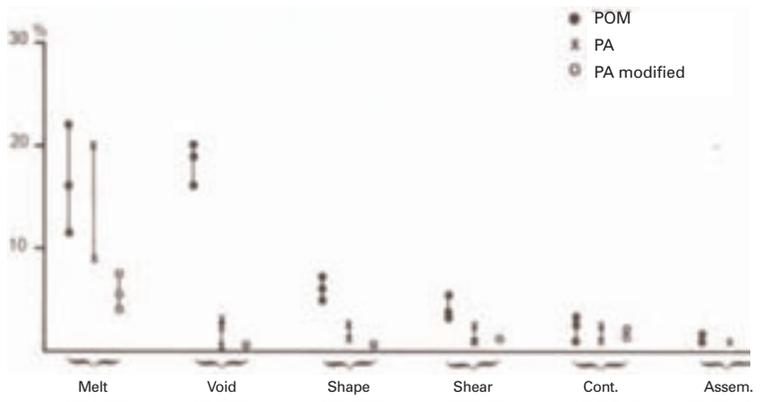
The reason for non-uniform structure, i. e. non-uniform crystallization can be:

- insufficient mixing of the melt in the barrel (which sometimes is more pronounced if a colour master batch is used);
- inadequate screw used for moulding barrel;
- a heater band on the moulding machine went out of function causing loss of heat for the passing melt at a certain area of the barrel;
- a combination of the above.

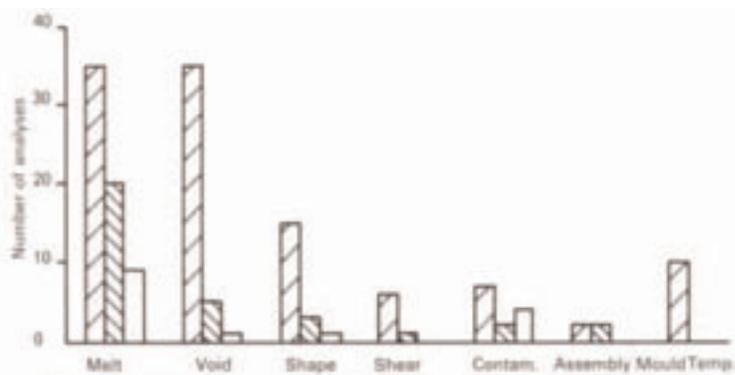
In all cases the mechanical properties suffer resulting in lower elongation, lower impact and increase in notch sensitivity compared to parts moulded at optimum processing conditions and thus having a uniform crystallinity growth.

It should be emphasized that these failures (unmelt and inhomogeneous melt) are the most frequent ones in failure analyses for parts moulded from crystalline resins (see graphs, p. 13 and 14).

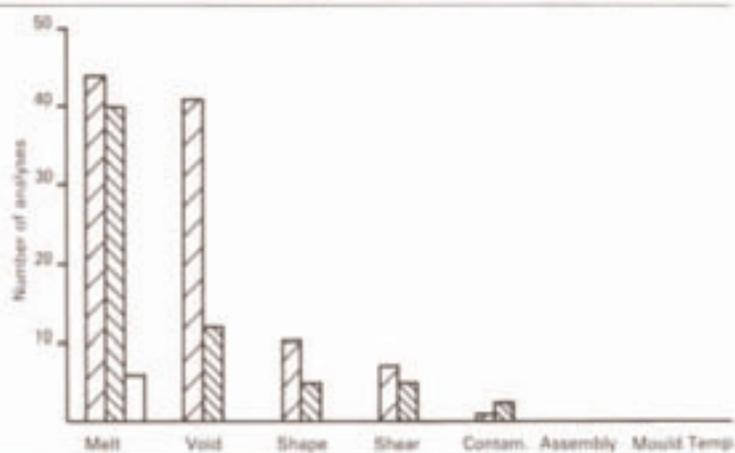
Type-of-Failure-Distribution
analysed by Structure Study
during 3 subsequent years.



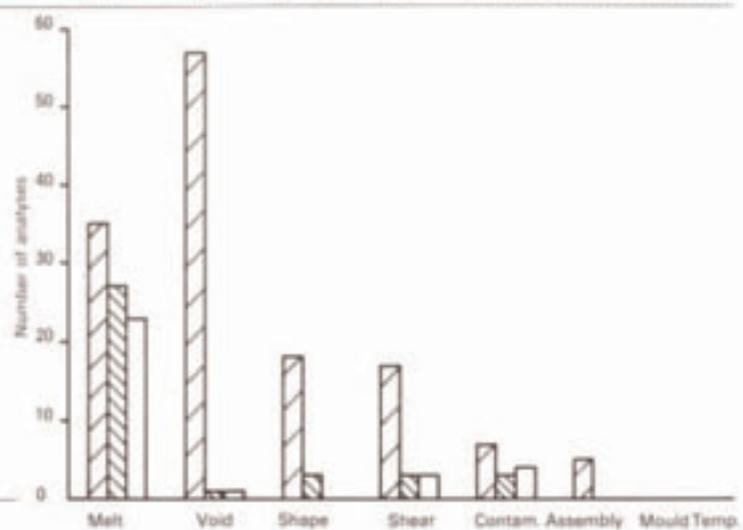
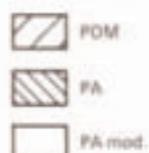
Year A



Year B



Year C



Weld Lines in Mouldings

Weld lines are almost inevitable when a moulded part has an insert, a boss, a hole, etc. Any insert or core is a sort of “obstacle” for the melt filling the cavity. The material flow is separated into two or more separate flows which will meet again to complete the cavity filling.

Assuming the melt temperature was already on the low side and hitting – in most cases – a cold insert, the material will not merge well when the separated flows meet. Thus a weld line becomes visible. Usually it can be seen on the surface of the part, almost always exactly opposite the gate location.

A visible weld line may not always be harmful as long as it does not extend throughout the total wall thickness. A microtomed section prepared perpendicular to the weld line will show how far the weld line goes through the cross section. In some cases it is a clear line – which may not always be a straight one, but can form different shapes, e.g. an “S”. The part in question is likely to have a reduced performance. Pressure, impact or other mechanical load in service may cause premature breakage which will usually occur along the weld line.

However, in some cases mechanical properties will not suffer from the presence of one or more weld lines as performance tests may reveal. Often only empirical evaluation will help to find the acceptable limits.

Mould Temperature

In general mould temperature affects mould shrinkage and dimensional stability of a part. It also influences mechanical properties, i. e. with increasing mould temperature stiffness increases and elongation at break decreases. Also, the hotter the mould the greater is the initial mould shrinkage and the better the dimensional stability after moulding. This is easily proven, e. g. on POM, by annealing a part after moulding.

The structure of the cross section of ASTM test bars moulded in DELRIN® (POM) under the same conditions but at different temperatures is shown in photo 1. The second crystallization zone is much thicker in the part moulded at the lowest temperature because this sample has a higher temperature gradient and faster cooling rate than those in the hot mould. However, the thickness of the second and center zone in relation to each other is influenced by the wall thickness of the part (see photo 3, page 18).

As other studies have shown, the centre zone which has the larger spherulites without preferred orientation, is the most stable one. It is desirable to mould polyacetals hot to obtain the stiffest and dimensionally most stable parts.

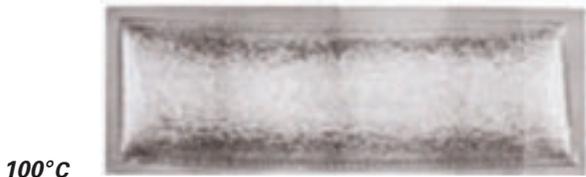
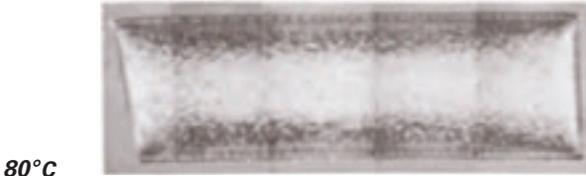
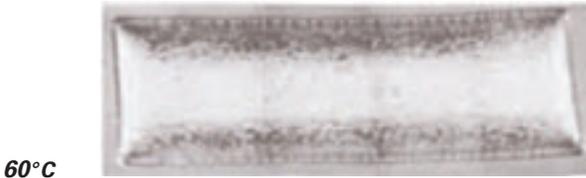
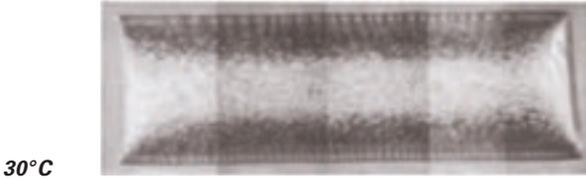
The structure of ZYTEL® (PA) is influenced as well (see photo 2) by mould temperature. However, its effect on mechanical properties is far less and is compensated by the moisture absorption at ambient atmosphere.

Influence of Mould Temperature on the Structure
 Microtome sections of moulded tensile bars (3,2 mm thickness)

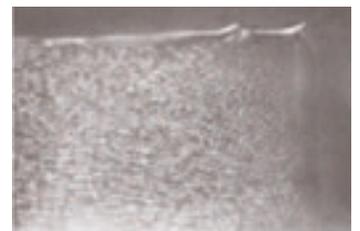
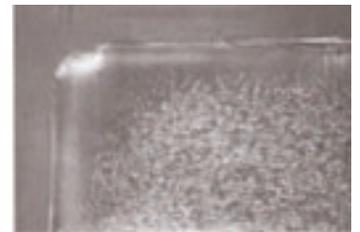
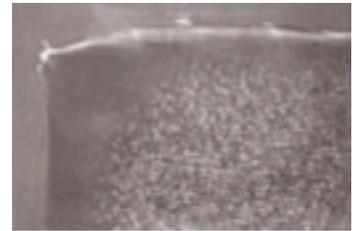
Mould Wall Temperature

Photo 1

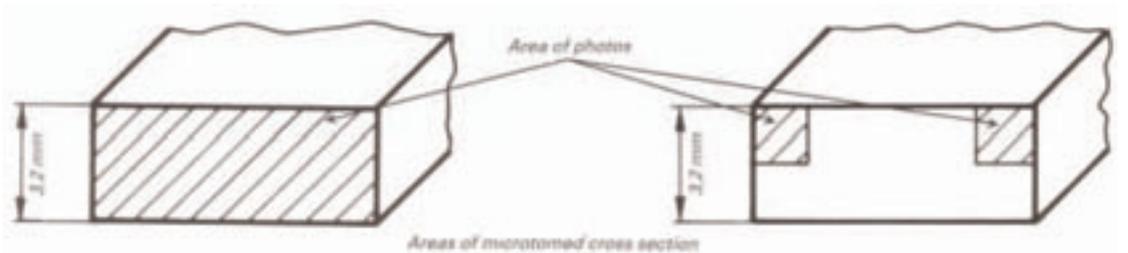
Photo 2



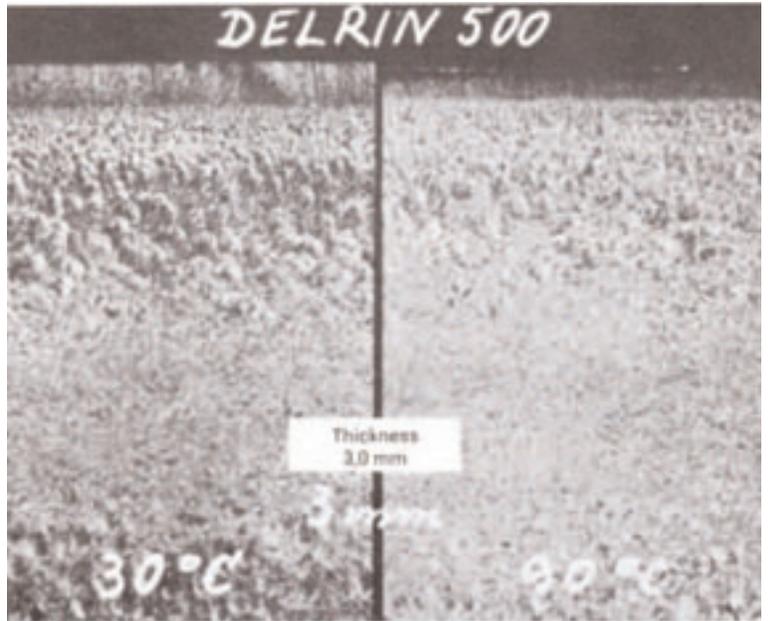
DELRIN® (POM)



ZYTEL® (PA 66)



60 ×



35 ×



Photo 3

Influence of mould temperature and wall thickness on the structure of DELRIN® 500 (Acetal homopolymer)

Screw Forward Time (SFT)



A = 25 sec. SFT



B = 15 sec. SFT



C = 5 sec. SFT

The screw forward time (injection time plus hold time) is certainly one of the most important factors in moulding parts for satisfactory performance. When the melt enters the mould, the material will freeze immediately along the mould wall and shrink. More material can then be pushed into the mould by maintaining the pressure until such time as the gate has frozen off. Thus it is obvious that gate design and area are also important factors. In case the SFT is not maintained until the gate freezes off, the sudden drop of pressure when the screw is retracted will influence the material due to the gate still being open. Pressure variations during screw forward time affect mould shrinkage, part weight and mechanical properties as well as crystal growth and configuration.

For example, with a tensile test bar of 3 mm thickness moulded of standard viscosity acetal homopolymer (DELTRIN® 500) the following phenomena occur with increasing SFT: decrease in mould shrinkage, increase in part weight and increase in elongation at break.

These effects are discussed (under ref. 5). Under optimal moulding conditions, i. e. if the SFT is maintained long enough until the whole material is frozen, proper mould shrinkage and better mechanical properties will result. The SFT is a function of the thickness of the part and the mould temperature. For a part with a thickness of 3 mm, such as an ASTM test bar, an SFT of 25 sec. should be sufficient at a mould temperature of 90°C.

The photo shows the cross sections of test bars of standard viscosity acetal homopolymer (DELTRIN® 500) moulded under the same conditions but one with 5 sec. SFT (C), one with 15 sec. SFT (B) and with 25 sec. SFT (A). In picture C a second line can be seen in the structure below the surface which appears all around the cross section. This line does not appear in the picture of the properly moulded part, and its presence can be explained as follows:

At the end of the 5 sec. SFT there is a sudden drop of injection pressure to zero and a correspondingly abrupt decrease in the freezing point. At this moment there is a sudden change of temperature gradient and hence of crystallization rate. This results in the formation of a new zone of orientation.

As the SFT is increased (B) the line of new orientation is much closer to the centre of the bar. This line moves closer to the centre as the SFT is increased and finally, it disappears completely. With a 25 sec. SFT for a 3 mm thick test bar we reach the point at which part weight is at maximum together with optimum values for all other properties.

These structural changes show why it is necessary to maintain injection pressure constant until the melt is completely frozen. Variations in pressure result in structural variations which can be noticed in the mechanical properties.

Shear Orientation

Shear orientation occurs when the molecules receive a preferred orientation while crystallizing. That is to a certain degree the case during the first phase of filling the cavity when the material freezes along the mould surface while still more melt is being injected. Thus the skin of a moulded part has a preferred orientation perpendicular to the surface. The high orientation prevents the growth of spherulites and in a thin section prepared from a moulded part the skin looks transparent (similar to amorphous material) when observed with polarized transmittant light.

The same kind of effect can be seen in the weld of a part welded by rotation or vibration because the material molten by high friction becomes oriented in the direction of rotation or vibration and freezes/crystallizes under that condition.

Shear orientation will also and inevitably occur at the injection point. How far this oriented zone reaches into the part can be influenced to some extent by the injection speed. This kind of shear orientation, however, does not result in a preferred order of crystallization and may cause internal stress. In cases where the part suffers impact or tension on such areas, mechanical strength will be reduced and premature breakage may occur.

Sharp corners due to too small a radius or bad mould manufacture can create a shear orientation as well causing premature breakage of the part.

Flash/Sharp Corner = Notch Effect

Flash on moulded parts can occur when either the clamping force of the moulding machine is insufficient or the viscosity of the molten resin is very low or the mould is abused and does not close correctly any longer.

In case a force is applied at the area of a part where flashing occurred the flash may break and cause a notch effect into the part which would not break otherwise.

Sharp corners may also cause a notch effect but for different reasons. Comparing thin sections of a moulded part having a sharp corner but not yet broken will reveal high stress concentration in that area while a rounded off corner does not show this effect. The high stress concentration will cause premature breakage under load.

The reasons can be:

- design not respecting the recommended minimum radius for moulded plastic parts;
- given radii had not been respected in tooling the mould;
- inserts or mould has been abused.

Voids

Large voids or areas of microporosity can be the result of insufficient filling of the cavity for various reasons such as:

- gate size too small and thus the material freezes off too early preventing injection of more material into cavity;
- filling thick wall sections through a thin wall by unfavourable location of the gate. Material of the thin section is already crystallized preventing sufficient filling of material into the thicker section;
- cycle too short and by subsequent shrinkage of the material voids will develop;
- material degraded during the moulding process. Consequently, development of gases prevents complete filling of the cavity.

Colour Agglomeration

In case a resin is not fully colour compounded, a masterbatch or liquid colouring is used to obtain the desired shades of colour. Only very rarely are powder pigments applied.

In all cases, uniform distribution of the pigment within the pigment carrier is necessary, otherwise not only the colour aspect is unsatisfactory. Additional to the visual effect a decrease in mechanical properties can be noticed. The pigment agglomeration reacts like a contaminant resulting in stress concentration and premature breakage.

Glass Fibre Orientation

Evaluation of the glass fibre orientation may sometimes help to explain differences in shrinkage and/or warpage observed in a part injection moulded from a glass reinforced crystalline material.

Microtome sections are not easy to obtain because the glass fibres may be torn out of the matrix resin while cutting. The polishing technique is recommended and microscopic inspection is often best using no filter at all.

With reflecting light and interference contrast, the orientation of glass fibres can also be observed on samples which have been printed and highly polished for the microscopic observation.

Once the main orientations and/or agglomerations of the glass fibres are recognized in the thin polished section prepared from the critical area of the part, moving the gate and/or changing the moulding parameters (e. g. injection speed) may alter the undesired behaviour of the moulded component.

Welding

Today, the welding of parts moulded in engineering thermoplastic resins is widely used as an assembly technique. Pressure tests can be used to check the strength of the weld, but the reason for a failure can only be detected by looking at the cross section of the weld area, or even better by examining a microtome section. In particular, microtome examination can be used to maintain control over weld time, weld pressure, dimensional precision and material condition.

Sometimes the microscopic examination reveals at the same time that the moulding conditions have not been at the optimum, but the mechanical strength of the weld may be within the limits of requirements. The evidence of crystallinity not being at its optimum might indicate which actions to take to improve the quality of the product further.

References

General

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- ⁶ Th. Speil: *Plastverarbeiter* 22, H.11 (1971), p. 785-792.
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- ¹⁰ Th. Speil: *Kunststoffe* 63, H.7 (1973), p. 463-468.
- ¹¹ E.S. Clark: *Plastic Engineering*, March (1974), p. 73-76.
- ¹² E. Böhme: *SPE – Techn. Paper* 20 (1974), p. 159ff.
- ¹³ E. Böhme: *Industrie-Anzeiger* 97, Jg. 23 (1975).
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- ⁱ H.U. Ischebeck:
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Practical metallography 21 (1984)

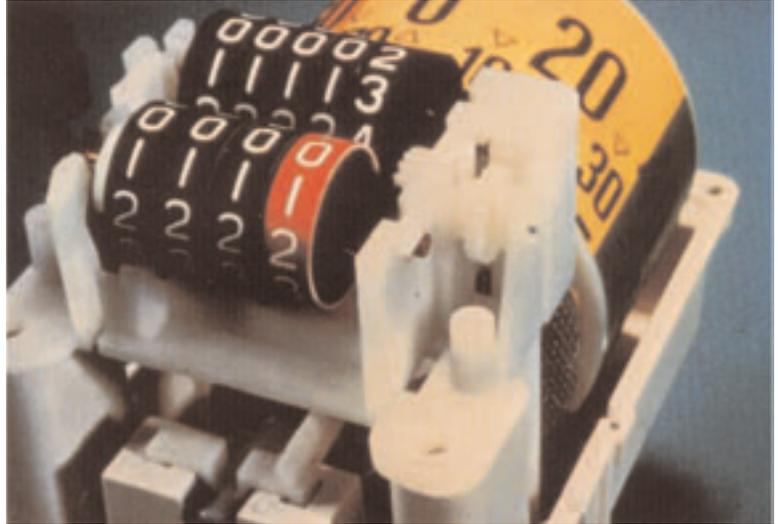
Inhomogeneous Melt

Part:
Counter wheels

Resin:
POM

Problem:
Occasional breakage of teeth in long term test series.

Reason for Failure:
Inhomogeneous melt increased brittleness and lowered resistance to impact load on start-up of gear.



A Parts

Microtome Cut	-			
Thin Polished Section	x			
Thickness	15 µm			
Microscopic Light				
Photo	A	B	C	D
Transmittant	-	x	x	
Pol. Filter	-	x	x	
Pol. Filter + Red Quartz	-	x	x	



M: 15



M: 22

B Inhomogeneous melt

C Enlargement of B

Inhomogeneous Melt

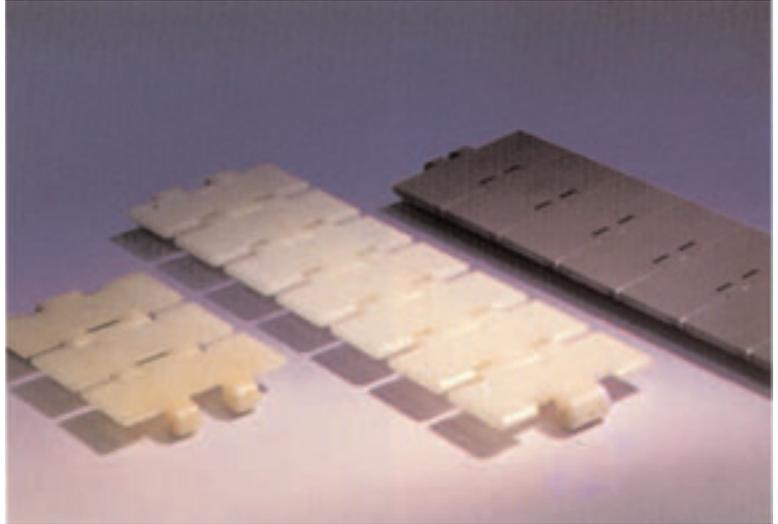
Part:
Conveyor chain

Resin:
POM

Problem:
Breakage during testing below specified load limits on some chain segments.

Reason for Failure:
Insufficient filling of cavity (voids) and inhomogeneous melt was reason for premature failure.

Remark:
The thick section should be redesigned to remove the tendency to form voids.



A Parts

Microtome Cut	×
Thin Polished Section	-
Thickness	25 µm
Microscopic Light	
Photo	A B
Transmittant	- ×
Pol. Filter	- ×
Pol. Filter + Red Quartz	- ×



M: 25

B Void and inhomogeneous melt

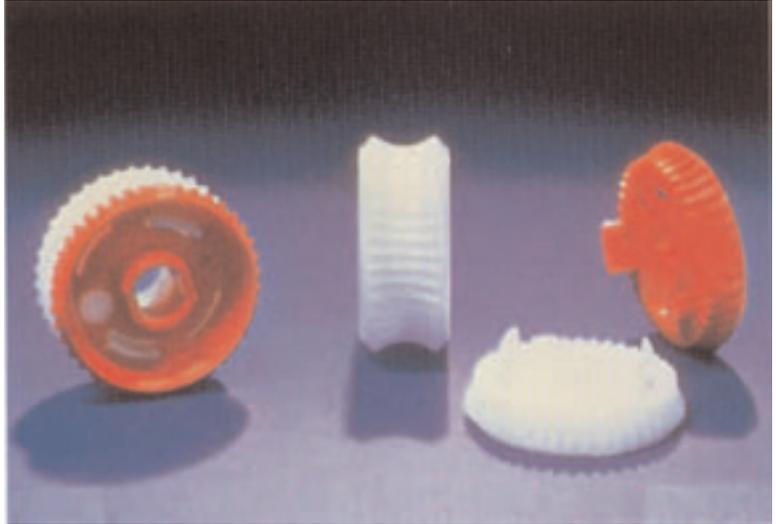
Inhomogeneous Melt

Part:
Gear, 45 mm Ø

Resin:
POM, natural

Problem:
Some teeth broke off in test at extremely low load.

Reason for Failure:
Unmolten particles due to too low melt temperature reducing strength of gear teeth.

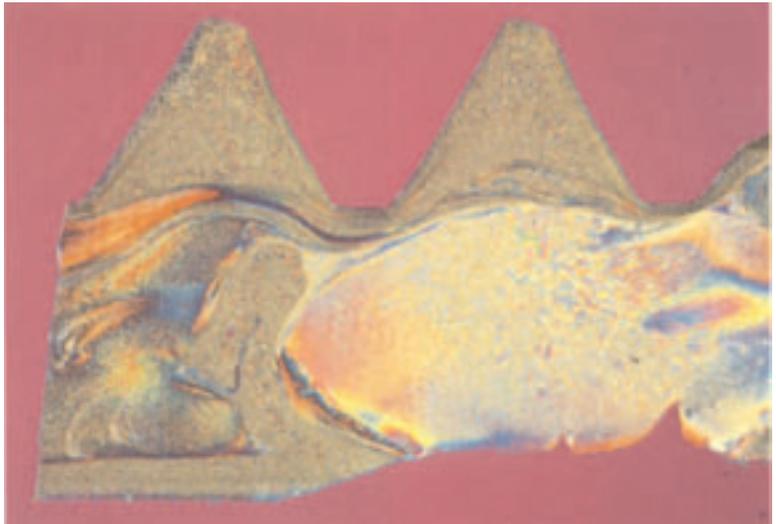


A Part

Microtome Cut	×
Thin Polished Section	-
Thickness	25 µm

Microscopic Light

Photo	A	B
Transmittant	-	×
Pol. Filter	-	×
Pol. Filter + Red Quartz	-	×



M: 20

B Break area

Inhomogeneous Melt

Part:
Socket, overmoulded

Resin:
POM, black

Problem:
Some parts in one delivery had whitish spots. As the POM was a blend of 1:1 natural with black resin, insufficient mixing in moulding process was assumed.

Reason for Failure:
Microtome cross section prepared from area of white spots revealed unmolten particles. These would cause premature failure.

Remark:
Additional determination of melting point from inspected area confirmed that white spots were the same POM as the homogeneous black resin.

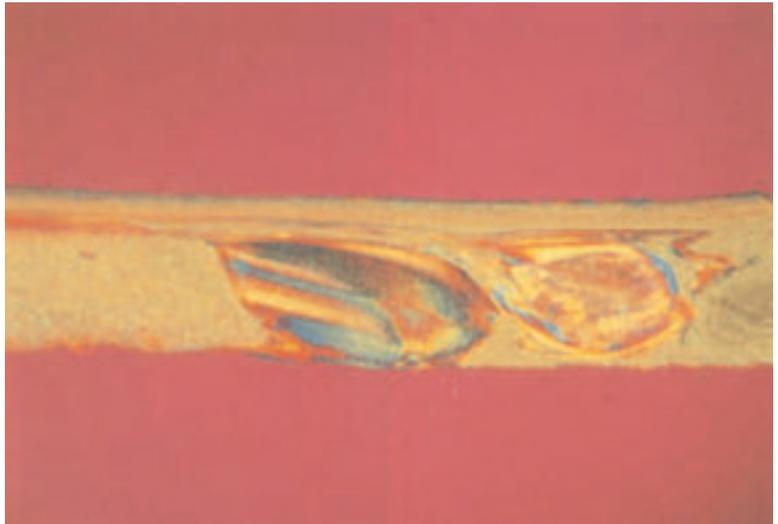


A Part

Microtome Cut ×
Thin Polished Section –
Thickness 25 µm

Microscopic Light

Photo	A	B
Transmittant	–	×
Pol. Filter	–	×
Pol. Filter + Red Quartz	–	×



M: 17

B Section from white spots

Inhomogeneous Melt

Part:
Bobbin for video tape

Resin:
POM

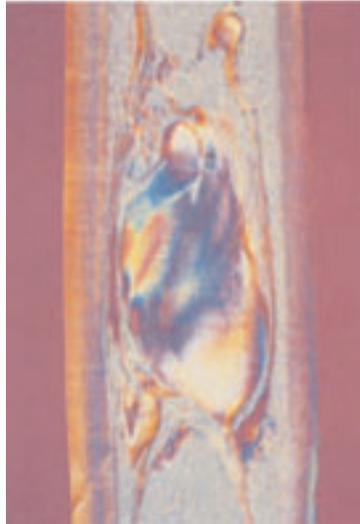
Problem:
In test series, whitish spots on surface and no dimensional stability from shot to shot.

Reason for Failure:
Both problems due to inhomogeneous melt.



A Part

Microtome Cut	x		
Thin Polished Section	-		
Thickness	20 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	-	x	x
Pol. Filter	-	x	x
Pol. Filter + Red Quartz	-	x	x



M: 22

B Inhomogeneous melt



M: 22

C Homogeneous melt

Inhomogeneous Melt

Part:
Safety clamping part

Resin:
POM, black

Problem:
Parts moulded of black and natural resin (blend 1:1) gave extremely low impact values in test. Break area reveals white spots.

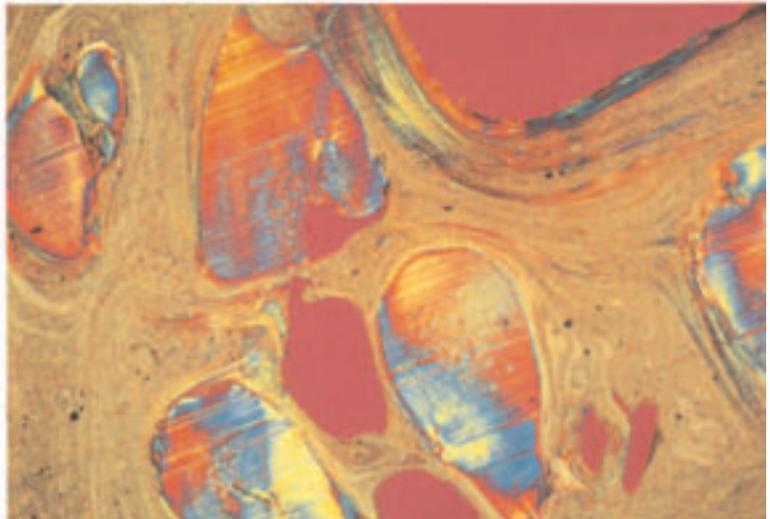
Reason for Failure:
White spots are particles of the natural resin which due to too low a melt temperature were not completely molten and thus acted like a contaminant reducing impact strength.

Remark:
In addition, parts are insufficiently filled resulting in voids.



A Part, break area

Microtome Cut	×
Thin Polished Section	–
Thickness	25 µm
Microscopic Light	
Photo	A B
Transmittant	– ×
Pol. Filter	– ×
Pol. Filter + Red Quartz	– ×



M: 33

B Unmolten particles and voids

Inhomogeneous Melt

Part:
Screw

Resin:
POM, black

Problem:
High reject rate in assembly line.
Heads of screws break off.
Cutting screw in half reveals void
(see arrow in photo A)
but voids are not at break area.

Reason for Failure:
Inhomogeneous melt and
sharp angle at first thread caused
brittleness.

Remark:
If the microtome cut or polished
section becomes **too thin**,
recognition of failure in structure
is not possible (see photo D).



A Part

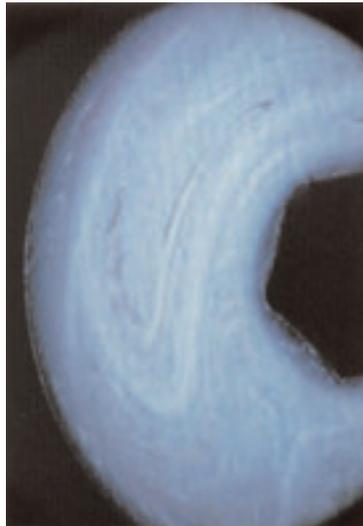
Microtome Cut -

Thin Polished Section ×

Thickness 20 μm

Microscopic Light

Photo	A	B	C	D
Transmittant	-	×	×	×
Pol. Filter	-	×	×	×
Pol. Filter + Red Quartz	-	-	-	-



M: 10

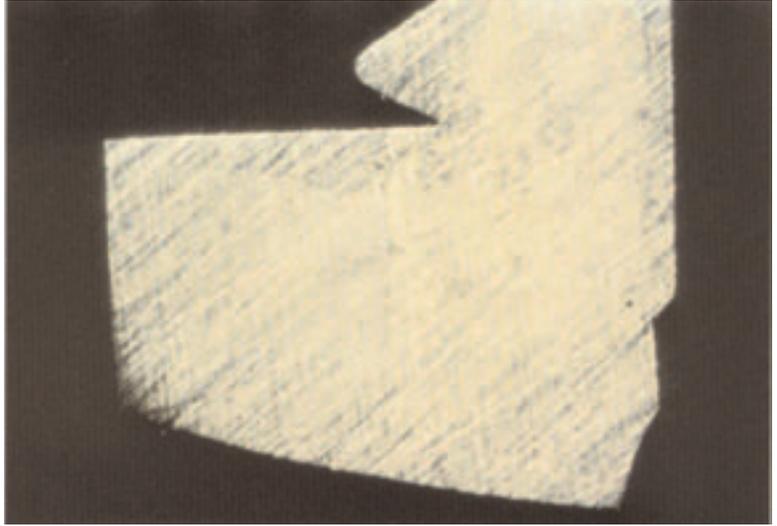
B Head, horizontal



M: 17

C Head, vertical

Inhomogeneous Melt



M: 25

D

As C, but too thinly polished

Inhomogeneous Melt

Part:

Gear with 3 springs

Resin:

POM, natural and grey

Problem:

After changing from natural to grey coloured material springs became brittle.

Reason for Failure:

Inhomogeneous melt in grey parts.

Remark:

Appearance of brittleness is independent of the change in colour and purely coincidental.



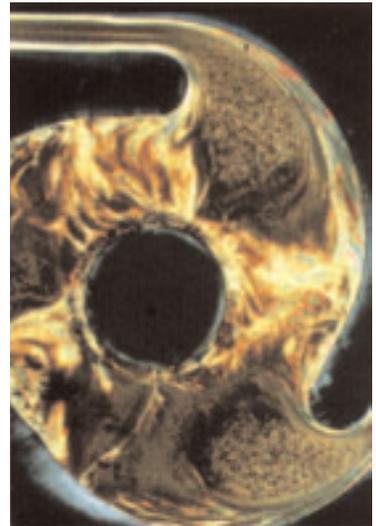
A Parts

Microtome Cut	-				
Thin Polished Section	x				
Thickness	20 µm				
Microscopic Light					
Photo	A	B	C	D	E
Transmittant	-	x	x	x	x
Pol. Filter	-	x	x	x	x
Pol. Filter + Red Quartz	-	-	-	x	x



M: 7

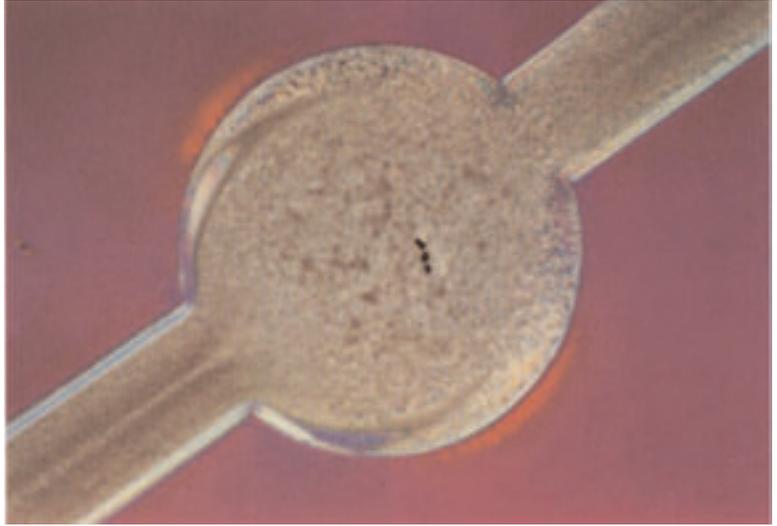
B Gear, natural – good



M: 7

C Gear, grey – brittle

Inhomogeneous Melt



M:40

D

Spring, natural – good



M:40

E

Spring, grey – brittle

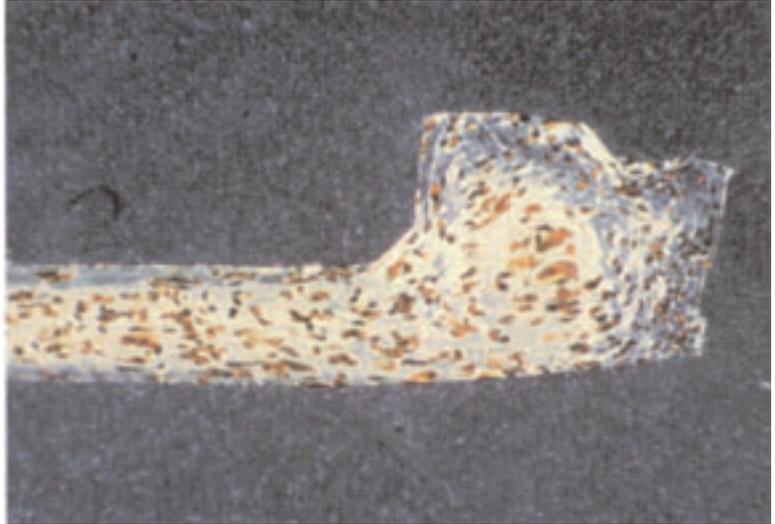
Inhomogeneous Melt

Part:
Bushing, overmoulded

Resin:
POM with PTFE-Fibres (20%)

Problem:
Overmoulding cracked under load.

Reason for Failure:
Inhomogeneous melt caused
brittleness.



M: 40

A Inhomogeneous melt

Microtome Cut	-
Thin Polished Section	×
Thickness	25 µm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	×
Pol. Filter + Red Quartz	-	-



M: 40

B Homogeneous melt

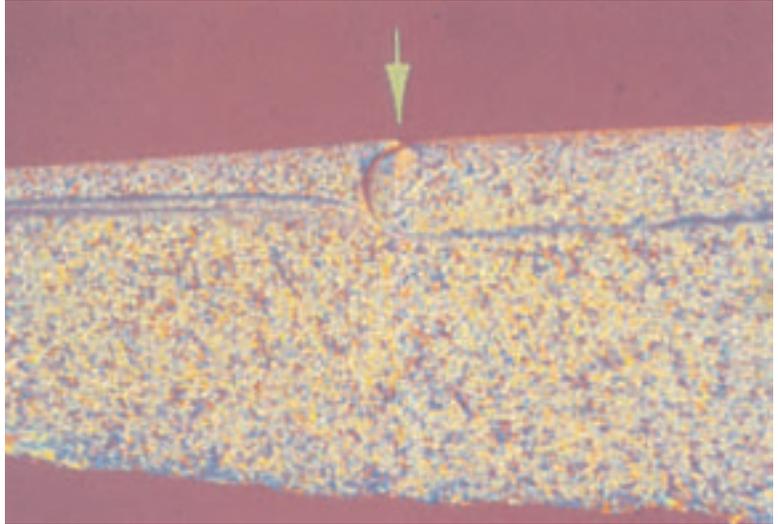
Inhomogeneous Melt

Part:
Refill bottle for lighter

Resin:
PA 66

Problem:
Quality control rejected parts showing surface defects easily detectable by naked eye.

Reason for Failure:
Cold thread from nozzle of moulding machine caused notch effect on surface (see arrow) emphasized by cold material underneath.



M: 20

A Thread of cold material near surface

Microtome Cut ×

Thin Polished Section –

Thickness 20 µm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz ×

Inhomogeneous Melt

Part:
Distance rings

Resin:
PA 66, coloured blue with water soluble dye

Problem:
No uniform colouring with one moulding series.

Reason for Failure:
Inhomogeneous melt prevented uniform absorption of dye.

Remark:
Speed of water absorption is influenced by size of spherulites. (Smaller spherulites = slower water absorption.) Thus non-uniform size of spherulites caused non-uniform absorption of water containing dissolved dye.



A Parts

Microtome Cut ×

Thin Polished Section -

Thickness 25 µm

Microscopic Light

Photo	A	B	C	D	E
Transmittant	-	×	×	×	×
Pol. Filter	-	×	×	-	-
Pol. Filter + Red Quartz	-	×	×	-	-



M: 17

B Inhomogeneous melt



M: 17

B Homogeneous melt

Inhomogeneous Melt



M: 25

D

Non-uniform absorption of dye



M: 25

E

Uniform absorption of dye

Inhomogeneous Melt

Part:

Container for break fluid

Resin:

PA 66

Problem:

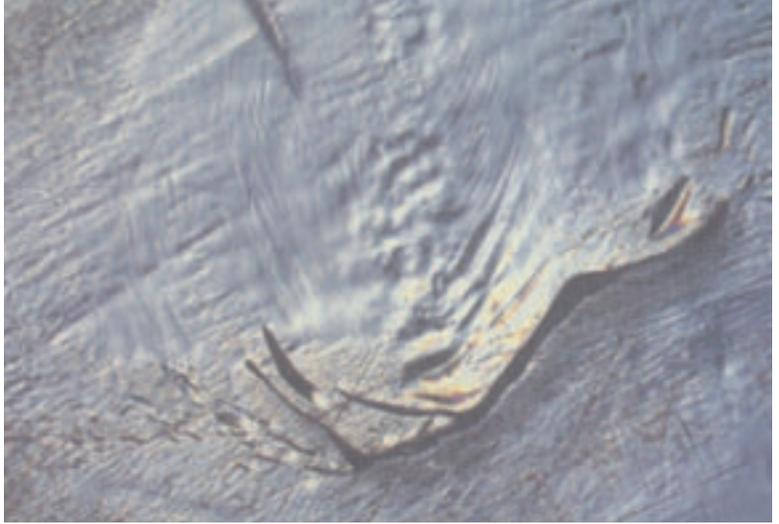
Brittleness in pressure test.

Reason for Failure:

Notch effect in areas where homogeneous and nonhomogeneous melt meet.

Remark:

Visible already on surface with naked eye.



M: 25

A Surface

Microtome Cut -

Thin Polished Section -

Thickness -

Microscopic Light

Photo A+B

Transmittant Reflecting Light

Pol. Filter Darkfield

Pol. Filter + Red Quartz -



M: 25

B Surface

Inhomogeneous Melt

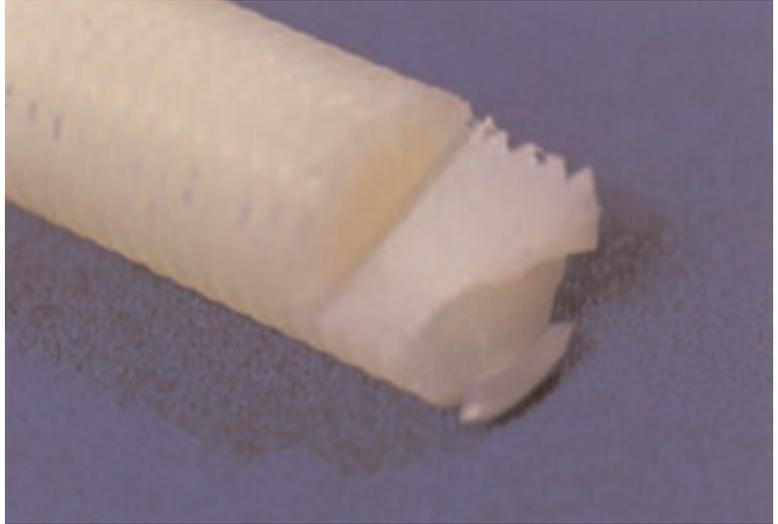
Part:
Extruded rod, 15 mm Ø

Resin:
PA 66, high viscosity resin

Problem:
Brittle breakage occurred when machining the threads.

Reason for Failure:
Unmolten particles in extruded material caused brittleness.

Remark:
The material giving no problem in machining showed a homogeneous structure.



A Rod with brittle break

Microtome Cut	×
Thin Polished Section	-
Thickness	25 µm

Microscopic Light

Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	×	×
Pol. Filter + Red Quartz	-	×	×



M: 21

B Cross section of A



M: 21

C Cross section of good rod

Inhomogeneous Melt

Part:
Ball bearing cage

Resin:
PA 66

Problem:
Series of delivery having varying performances:
– series likely to be distorted
– series having strong distortion so that balls are falling out
– good series from earlier delivery

Reason for Failure:
Series of decreasing quality show decrease in homogeneity of structure, e. g. melt temperature was getting lower from one series to the next.

Remark:
Series with strong distortion show in addition weld line due to too low a melt temperature.



A Parts



M: 14

B Likely to be distorted

Microtome Cut ×

Thin Polished Section –

Thickness 20 µm

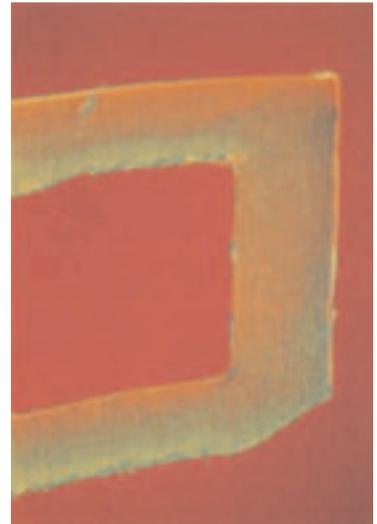
Microscopic Light

Photo	A	B	C	D
Transmittant	–	×	×	×
Pol. Filter	–	×	×	×
Pol. Filter + Red Quartz	–	×	×	×



M: 14

C Strong distortion



M: 14

D No distortion

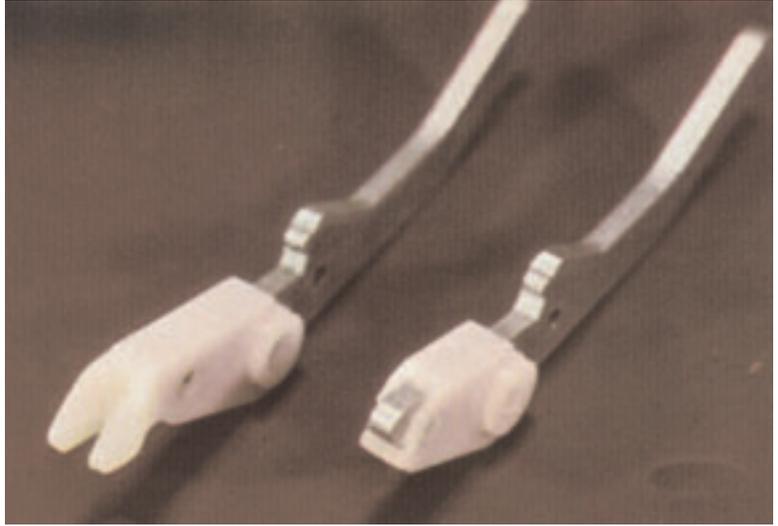
Inhomogeneous Melt

Part:
Lever

Resin:
PA 66, heat stabilized

Problem:
Breakage of parts in use.

Reason for Failure:
Inhomogeneous melt reduced impact strength and elongation.

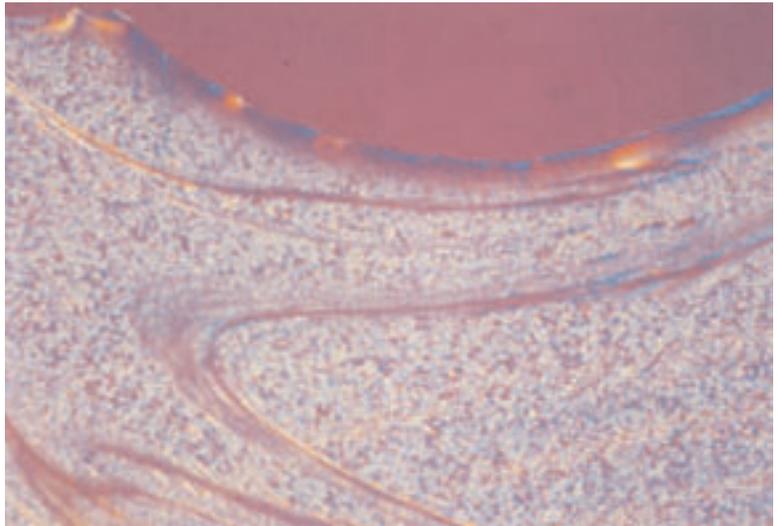


A Part

Microtome Cut	×
Thin Polished Section	-
Thickness	25 μm

Microscopic Light

Photo	A	B
Transmittant	-	×
Pol. Filter	-	×
Pol. Filter + Red Quartz	-	×



M: 25

B Inhomogeneous melt

Inhomogeneous Melt

Part:
Sealing ring

Resin:
PA 66

Problem:
Snap-fit of sealing ring was too soft
and did not hold tight.

Reason for Failure:
In comparison to the rings which
performed satisfactorily the soft
rings had a lower crystallinity
(more transparent appearance).
Thus stiff-ness was reduced.



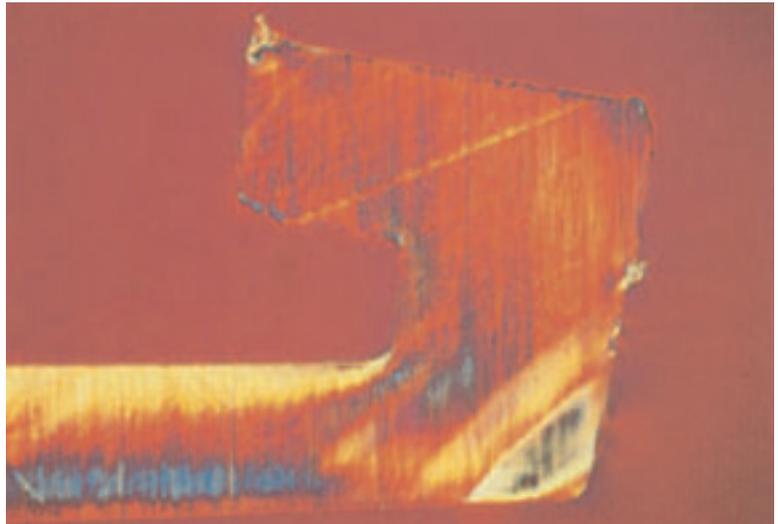
M: 50

A High crystallinity

Microtome Cut	-
Thin Polished Section	×
Thickness	15 μm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	×
Pol. Filter + Red Quartz	×	×



M: 50

B Low crystallinity

Inhomogeneous Melt

Part:
Standard impact bar
(Charpy 50 × 6 × 4 mm)

Resin:
PA 66, modified

Problem:
High variation in impact strength on
standard bars moulded at customer.

Reason for Failure:
Poor melt quality and low melt
temperature caused irregular impact
resistance.

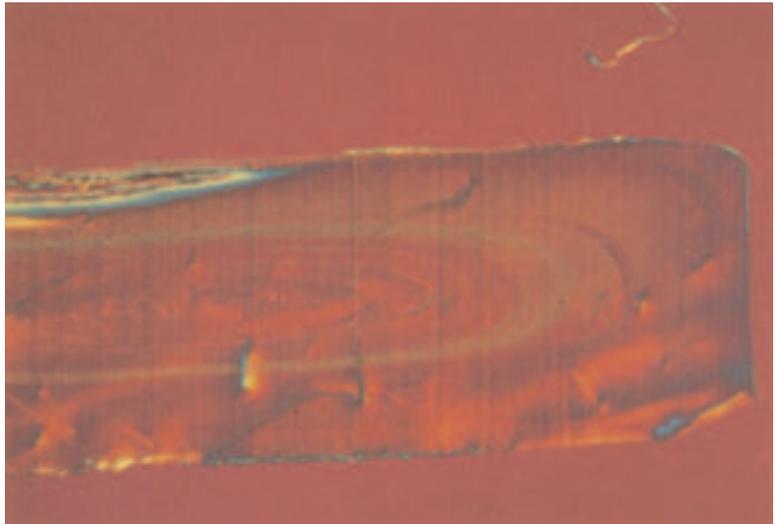
Remark:
As proof, impact bars were moulded
with correct and too low melt
temperature. Failure could be
reproduced. Moulding parameters
other than cold melt are difficult
to recognize in the structure of
modified PA.



M: 20

A Correct melt

Microtome Cut	×
Thin Polished Section	–
Thickness	20 μm
Microscopic Light	
Photo	A B
Transmittant	×
Pol. Filter	×
Pol. + Red Quartz	×



M: 20

B Inhomogeneous melt

Inhomogeneous Melt

Part:

Cable strap, different types

Resin:

PA 66, natural and black

Problem:

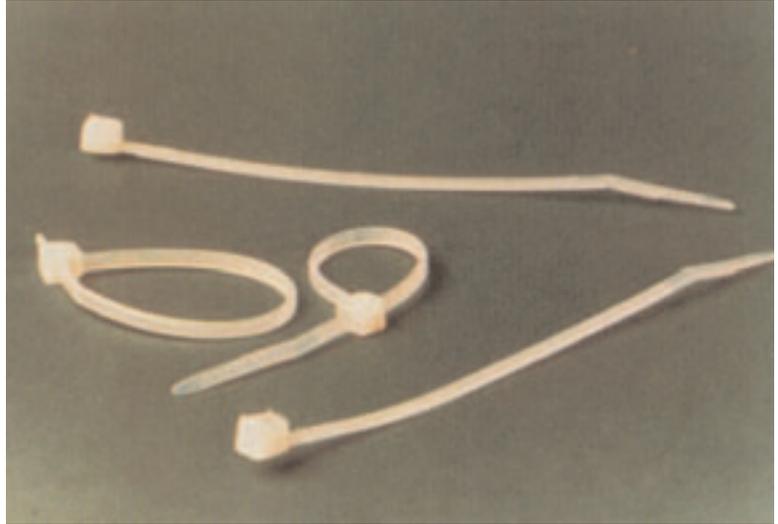
Brittle break in bending test or in assembly.

Reason for Failure:

Inhomogeneous melt. In some cases sharp corner in tooth base. One has high shear orientation around the base of the teeth. This shear might be caused by too low melt temperature (D).

Remark:

Sharp corners in the base of the teeth cause stress concentration and notch effect resulting in premature break under load.



Microtome Cut	-	
Thin Polished Section	x	
Thickness	20 µm	

Microscopic Light

Photo	A	B to J
Transmittant	-	x
Pol. Filter	-	x
Pol. Filter + Red Quartz	-	x

A

Part



M: 21



M: 28

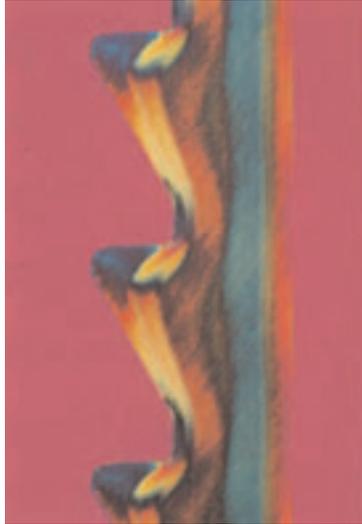
B

Inhomogeneous melt

C

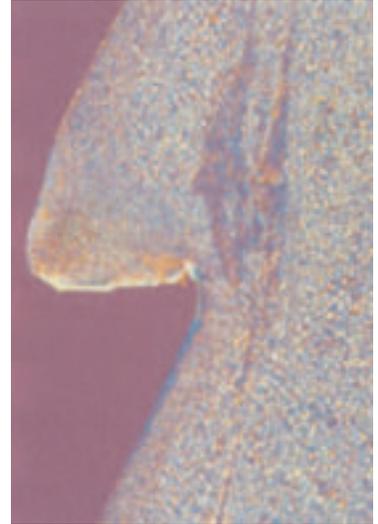
Homogeneous melt

Inhomogeneous Melt



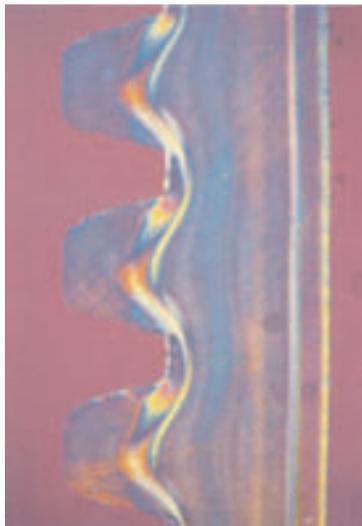
M: 17

D Shear along teeth



M: 55

E Inhomogeneous melt



M: 9

F Inhomogeneous melt

Inhomogeneous Melt



M: 7



M: 7

G Inhomogeneous melt

H Homogeneous melt



M: 22



M: 22

I Enlarged section of G

J Enlarged section of H

Inhomogeneous Melt

Part:
Standard specimens (tensile bars)

Resin:
PA 66 with mineral (40%)

Problem:
Evaluation of possibility to recognize low or inhomogeneous melt in 66-nylon containing mineral.

Reason for Failure:
Standard tensile bars (3,2 mm thickness) moulded with correct and 20°C lower melt temperature reveal evidence of cold melt in thin section.



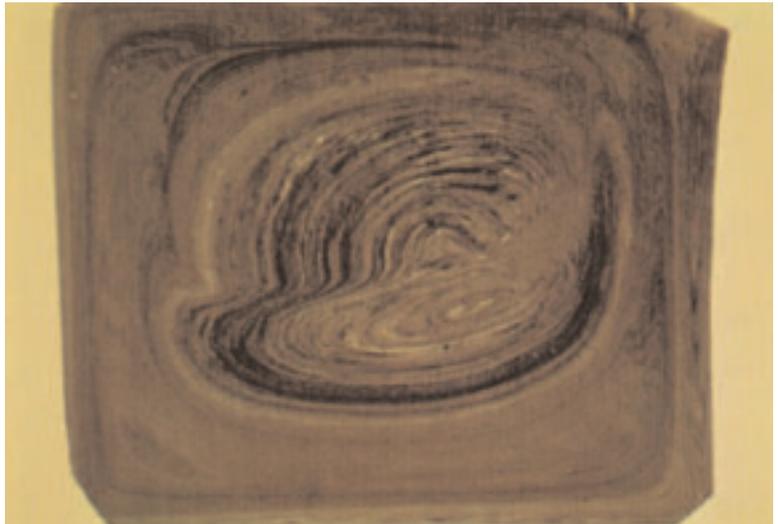
M: 25

A Correct melt temperature

Microtome Cut	×
Thin Polished Section	-
Thickness	20 µm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	30°
Pol. Filter + Red Quartz	-	-



M: 20

B Inhomogeneous melt

Weld Line

Part:

Gear, 4 mm Ø, 6 mm height

Resin:

POM

Problem:

Some teeth break off in test run (gear has 2 injection points).

Reason for Failure:

Weld line due to too cold melt temperature when melt flows meet. In some gears the weld line is located at a tooth base angle (D).



A Part



B Thinly polished gears

Microtome Cut -

Thin Polished Section ×

Thickness 20 µm

Microscopic Light

Photo	A	B	C	D
Transmittant	-	×	×	×
Pol. Filter	×	×	×	×
Pol. Filter + Red Quartz	-	-	×	-



M: 9

C Weld line between two teeth



M: 11

D Weld line at tooth base angle

Weld Line

Part:
Trim clip

Resin:
POM

Problem:
Parts break during assembly.
Break area almost opposite to gate.

Reason for Failure:
Weld line formed on surface only due to too fast cooling at mould surface when melt flows around the core. Thus notch effect.

Remark:
Weld line does not appear throughout the whole cross section. Therefore notch effect from surface only when stressed during assembling (see area between arrows on photo D).



A Trim clip



B Weld line marked with soft pencil

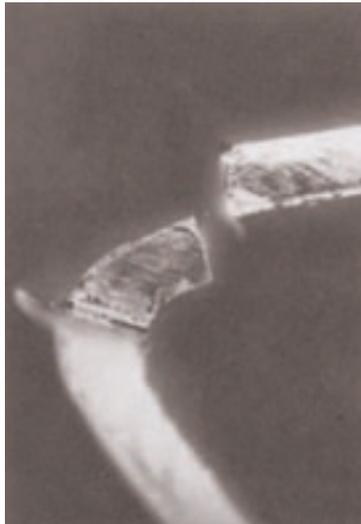
Microtome Cut -

Thin Polished Section × for D

Thickness 10 μm

Microscopic Light

Photo	A	B	C	D
Transmittant	-	-	-	×
Pol. Filter	-	-	-	×
Pol. Filter + Red Quartz	-	-	-	×



M: 7

C Break at marked area



M: 7

D Cross section at marked area

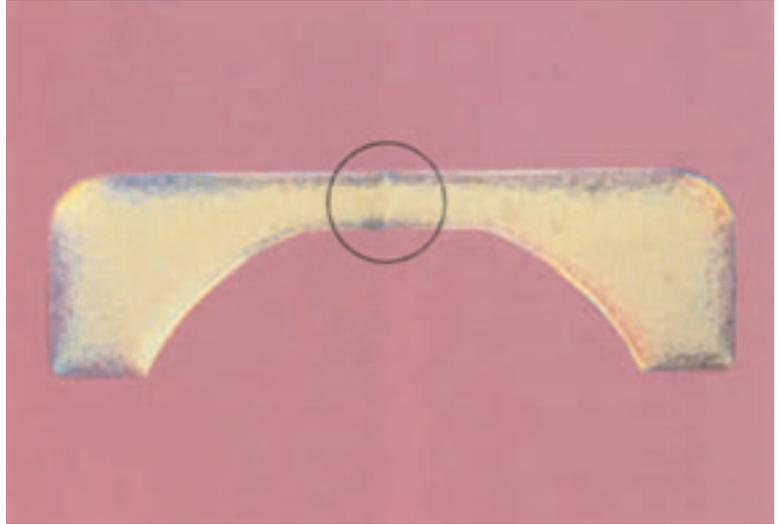
Weld Line

Part:
Functional part

Resin:
POM

Problem:
Part of the production gave breakage below threshold limit in quality control testing.

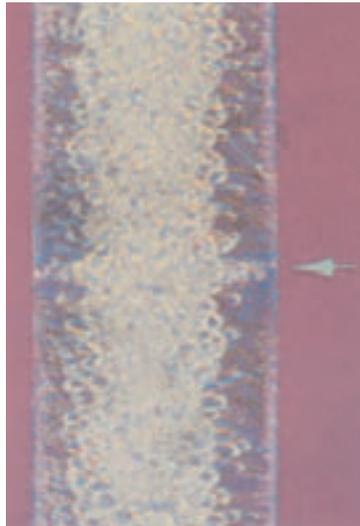
Reason for Failure:
In comparison to parts within limits of test specification, the failing parts show a distinct weld line in cross section.



M: 9

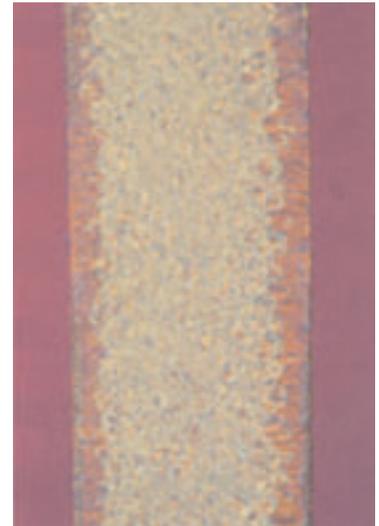
A Weld line

Microtome Cut	-		
Thin Polished Section	x		
Thickness	20 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	x	x	x
Pol. Filter	x	x	x
Pol. Filter + Red Quartz	x	x	x



M: 44

B Enlarged area of weld line



M: 44

C Same cross section as B from good part

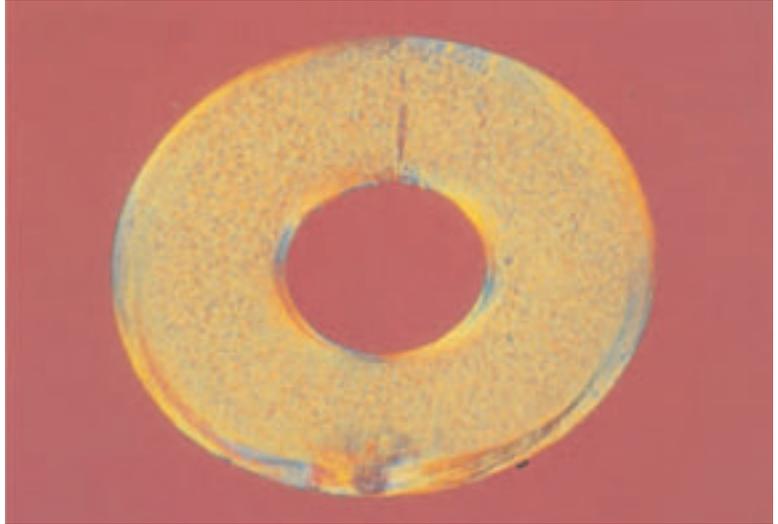
Weld Line

Part:
Bushing

Resin:
POM

Problem:
Bushings crack opposite to gate when forced onto axle.

Reason for Failure:
Melt has been cooled off too much while flowing in cavity around the core preventing a homogeneous weld. Reason can be too low melt temperature or too slow injection speed.



M: 25

A Weld line

Microtome Cut ×

Thin Polished Section –

Thickness 20 μm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz ×

Weld Line

Part:

Plate with threaded boss

Resin:

POM/POM, modified (blend)

Problem:

Part cracked at weld line when stud was screwed in. Notch effect on surface (see arrow).

Reason for Failure:

Weld line in structure goes through the whole cross section. Weld line appears when melt is already too cold and material flow meets behind the core.

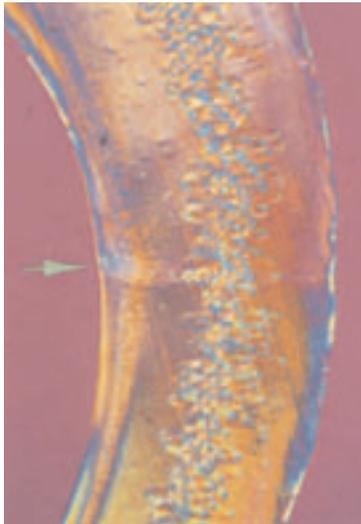


A Part showing crack

Microtome Cut	×
Thin Polished Section	-
Thickness	20 μm

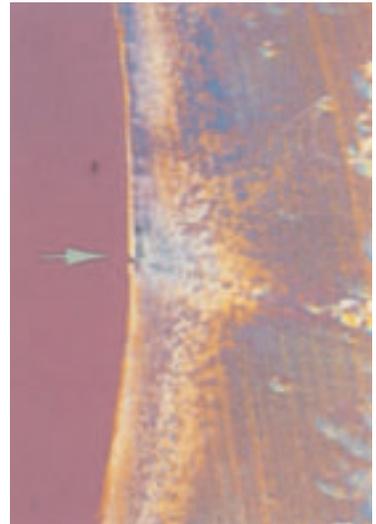
Microscopic Light

Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	×	×
Pol. Filter + Red Quartz	-	×	×



M: 17

B Weld line in cross section



M: 44

C Enlarged area of B

Weld Line

Part:
Window handle

Resin:
POM, coloured with masterbatch

Problem:
Parts break in test.

Reason for Failure:
Position of gate in relation to load direction not optimum. In addition, low melt temperature caused weld line.

Remark:
The low melt temperature also prevented homogeneous dispersion of masterbatch.



A Parts

Microtome Cut	×
Thin Polished Section	-
Thickness	25 µm

Microscopic Light

Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	×	×
Pol. Filter + Red Quartz	-	-	-



M: 7



M: 17

B Weld line and inhomogeneous melt

C Enlarged, weld line

Weld Line

Part:
Bushing

Resin:
PA 66, high viscosity resin

Problem:
Breakage at loads under the specified limits. Breaks occurred opposite gate.

Reason for Failure:
Weld line clearly visible in thin section due to too low melt temperature.



M: 10

A Low melt temperature = weld line

Microtome Cut	-
Thin Polished Section	×
Thickness	15 μm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	×
Pol. + Red Quartz	×	×



M: 10

B Homogeneous melt

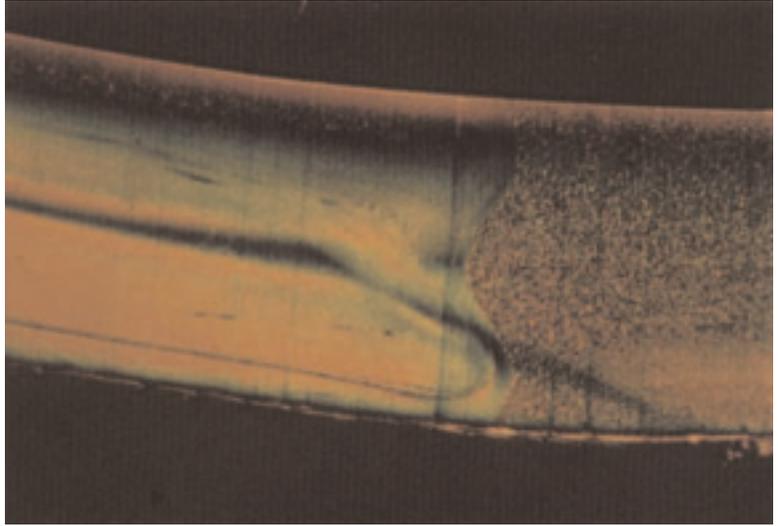
Weld Line

Part:
Bushing

Resin:
PA 66, unreinforced

Problem:
High reject rate in pressure test.
Position of break seemed to be
at weld line.

Reason for Failure:
Two reasons, both due to too low
melt temperature.
a) weld line (in S-shape)
b) partially finer structure similar to
nucleated PA. The latter has lower
elongation in dry-as moulded
condition than normal structure



M:40

A Weld line

Microtome Cut ×
perpen-
dicular
to flow

**Thin Polished
Section** –

Thickness 20 µm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz ×

Weld Line

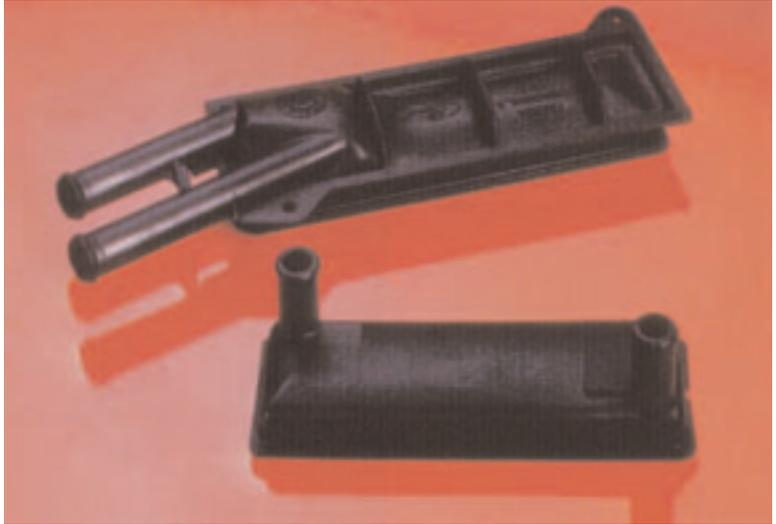
Part:
Radiator tanks

Resin:
PA 66, 30% glass reinforced

Problem:
Weld line opposite to gate was visible to naked eye. Quality of weld checked by mechanical tests (Dynstat impact strength) and thin polished section.

Reason for Failure:
No failure observed. Although weld line is visible in thin polished section, no loss in impact strength compared to other areas of same part.

Remark:
S-shape of weld line in glass reinforced PA seems to give good mechanical strength. Best observation is without any filter.



A Part

Microtome Cut	-			
Thin Polished Section	x			
Thickness	15 µm			
Microscopic Light				
Photo	A	B	C	D
Transmittant	-	x	x	x
Pol. Filter	-	-	x	x
Pol. Filter + Red Quartz	-	-	-	x



M:12

B Weld line



M:12

C Weld line

Weld Line



M:17

D Weldline

Weld Line

Part:
Spoke wheel

Resin:
PA 66, modified, 15% glassfiber content

Problem:
Control of weld line between two gates. Break in test at weld line.

Reason for Failure:
Presence of weld line can not be eliminate. In this case impact tests confirmed sufficient mechanical strength.

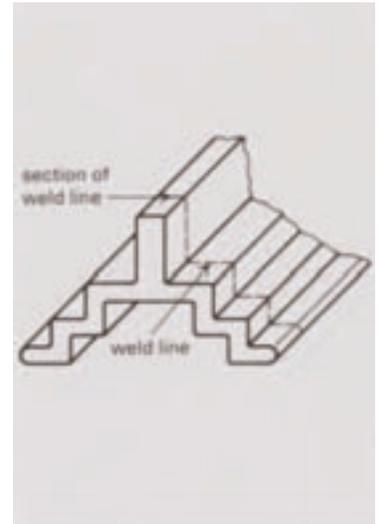


A Wheels

Microtome Cut	-		
Thin Polished Section	x		
Thickness	30 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	-	x	-
Pol. Filter	-	-	-
Pol. Filter + Red Quartz	-	-	-



M:28



B Weld line

C Weld line – Schema

Mould Temperature

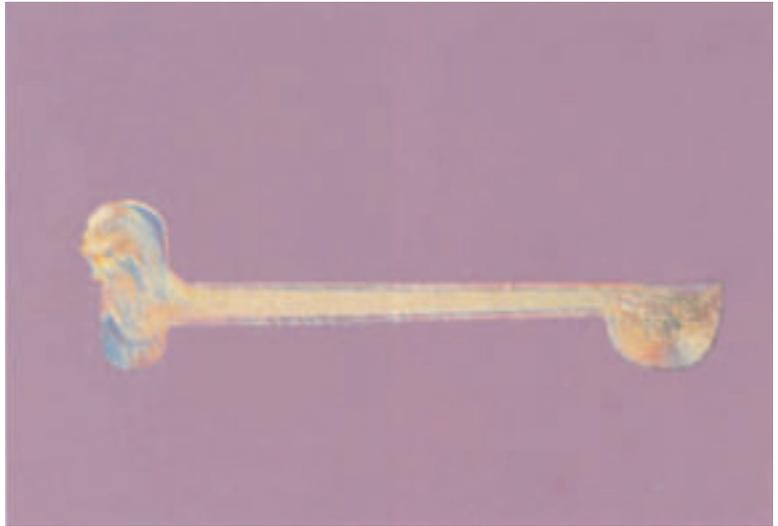
Part:
Spring

Resin:
POM

Problem:
In long term testing a few springs showed less fatigue resistance than other ones.

Reason for Failure:
Too low mould temperature of failing springs, as seen by comparison of cross sections, resulted in a more flexible part explaining differences.

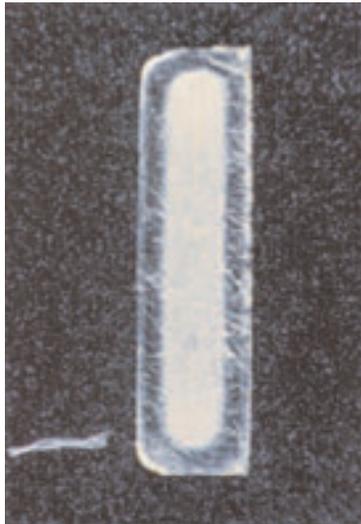
Remark:
Systematic studies on effect of mould temperature on mechanical properties of POM-homopolymer have explained different behaviour (see literature).



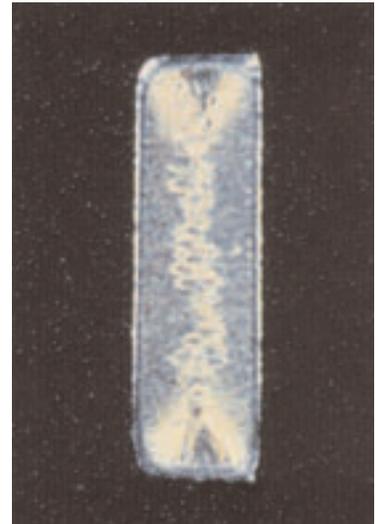
M: 10

A Part

Microtome Cut	-		
Thin Polished Section	x		
Thickness	15 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	x	x	x
Pol. Filter	x	x	x
Pol. Filter + Red Quartz	x	-	-



M: 35



M: 35

B Low mould temperature

C Correct mould temperature

Mould Temperature

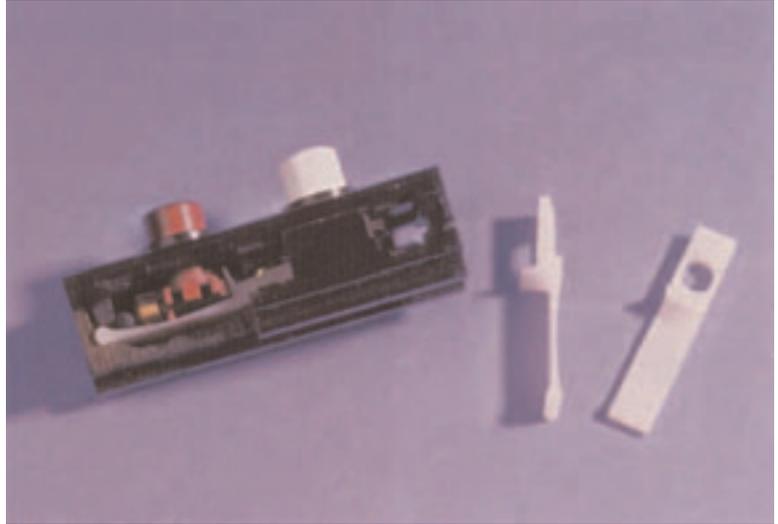
Part:
Spring in light switch

Resin:
POM

Problem:
Prototype **machined** from extruded rod performed 180000 cycles without fatigue. Same part **moulded** resisted in test 80000 cycles only.

Reason for Failure:
Machined part consisted of highly crystalline material with big spherulites without preferred orientation. In comparison the moulded part shows 3 different zones of orientation of the spherulites resulting in lower stiffness/higher elasticity thus less fatigue resistance.

Remark:
See literature ref. 5.



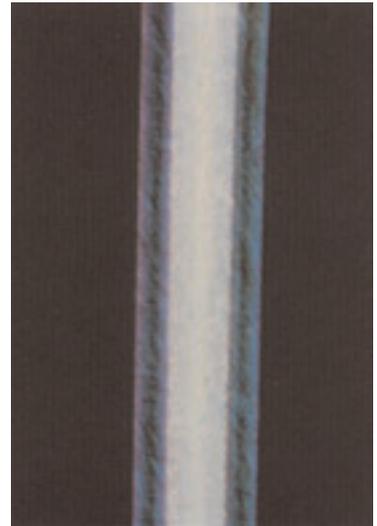
M: 10

A Part

Microtome Cut	-		
Thin Polished Section	x		
Thickness	15 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	-	x	x
Pol. Filter	-	x	x
Pol. + Red Quartz	-	-	-



M: 11



M: 11

B Machined prototype

C Moulded prototype

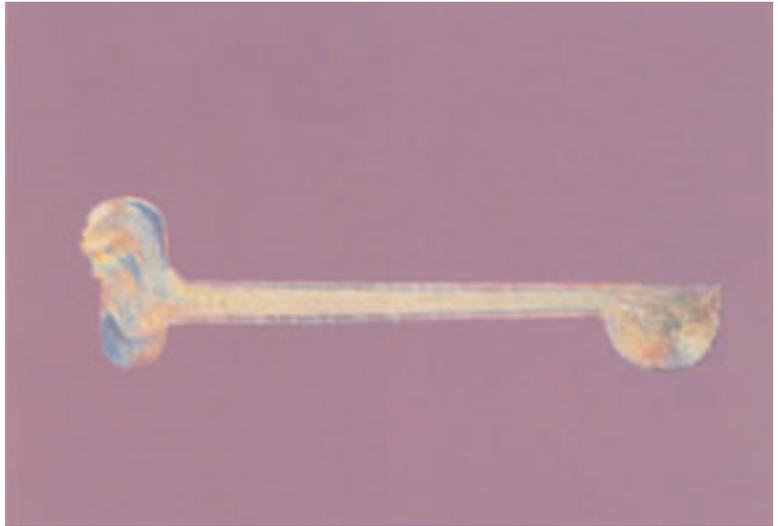
Screw Forward Time

Part:
Spring

Resin:
POM

Problem:
In test series some of the parts had less fatigue resistance than other ones.

Reason for Failure:
Structure shows line of too short SFT, reducing mechanical properties.



M: 10

A Part

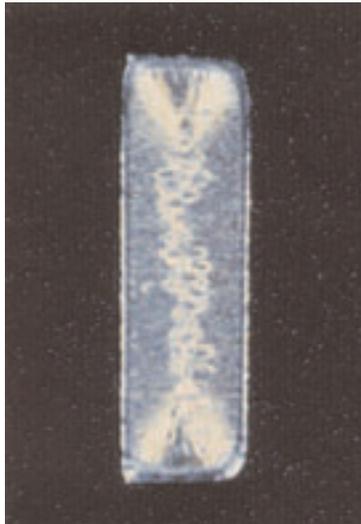
Microtome Cut -

Thin Polished Section ×

Thickness 15 μm

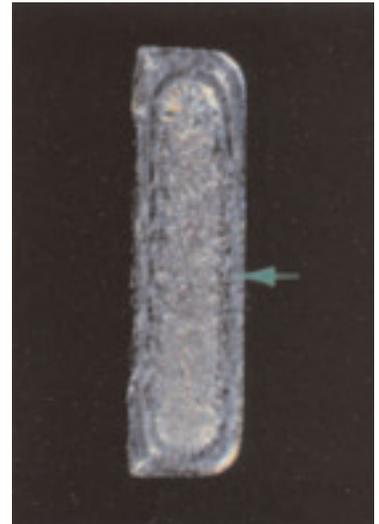
Microscopic Light

Photo	A	B	C
Transmittant	×	×	×
Pol. Filter	×	×	×
Pol. Filter + Red Quartz	×	-	-



M: 35

B Cross section of good part



M: 35

C Cross section showing SFT-line

Voids

Part:
Zipper

Resin:
POM

Problem:
Teeth break off.

Reason for Failure:
Insufficient filling of mould cavity resulting in voids. Thus, reduced cross section gives lower mechanical strength.



M: 17

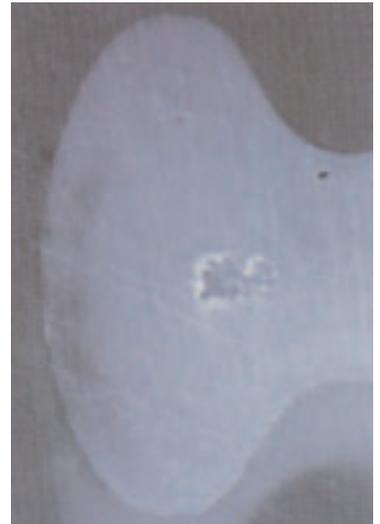
A Zipper

B Cross section

Microtome Cut	-			
Polished Section	Reflectant light Darkfield			
Thickness	-			
Microscopic Light				
Photo	A	B	C	D
Transmittant	-	-	-	-
Pol. Filter	-	-	-	-
Pol. Filter + Red Quartz	-	-	-	-



M: 17



M: 28

C Section lengthwise

D Section lengthwise

Shear Orientation

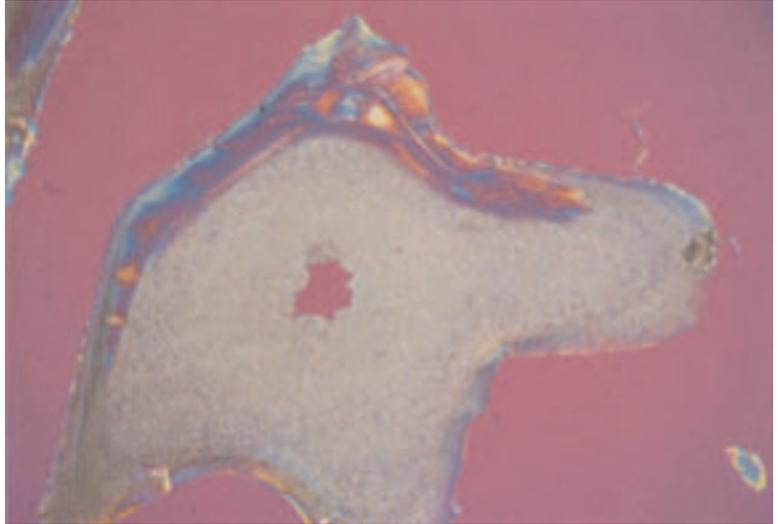
Part:
Snap ring for eyepiece of binocular

Resin:
POM, black

Problem:
Ring breaks at snap fit area (100% reject).

Reason for Failure:
Strong shear orientation at gate (photo A) reduced elongation of ring. Slower injection speed reduced shear orientation considerably to produce acceptable function of the parts.

Remark:
Failing parts were also not well filled showing big voids in center. Thus the functional cross section was reduced too.



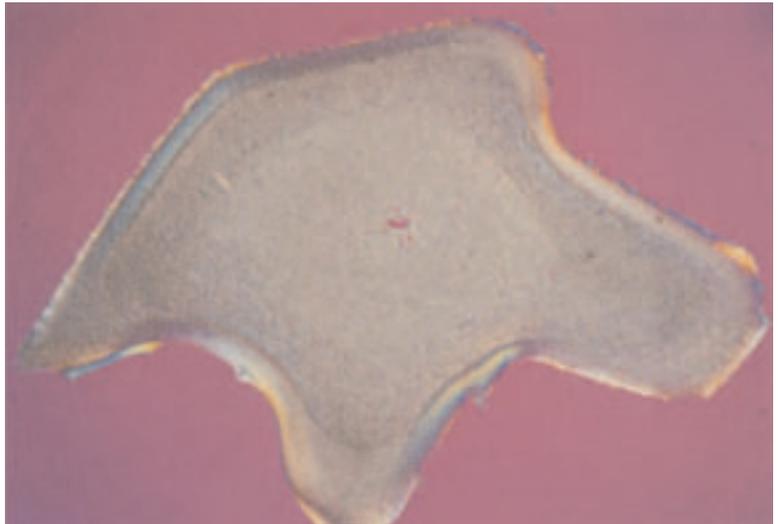
M: 13

A Strong shear at gate

Microtome Cut	×
Thin Polished Section	–
Thickness	20 μm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	×
Pol. Filter + Red Quartz	×	×



M: 13

B Reduced shear at gate

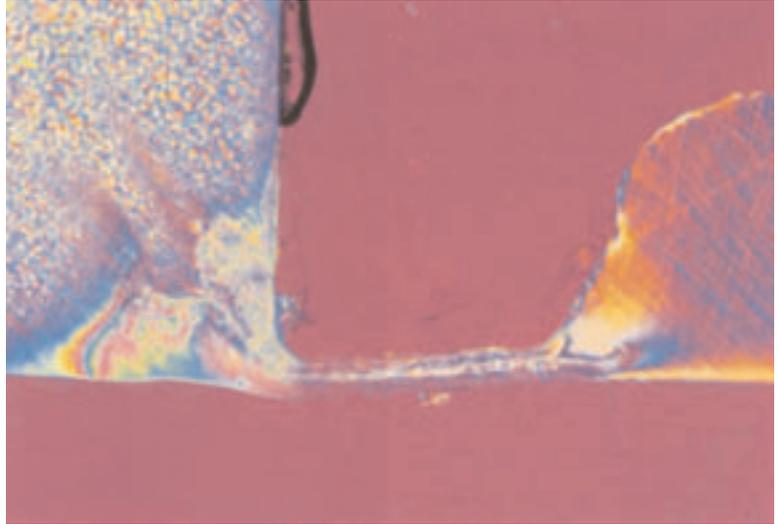
Shear Orientation

Part:
Seal

Resin:
PA 66

Problem:
No problem, but control of desired break area designed on purpose to act as "safety valve".

Reason for Failure:
Shear orientation is clearly seen at corners of thin wall cross section. The reduced strength perpendicular to the orientation results in breakage in the desired area.



M: 80

A Shear orientation

Microtome Cut	-
Thin Polished Section	×
Thickness	10 μm
Microscopic Light	
Photo	A
Transmittant	×
Pol. Filter	×
Pol. Filter + Red Quartz	×

Shear Orientation

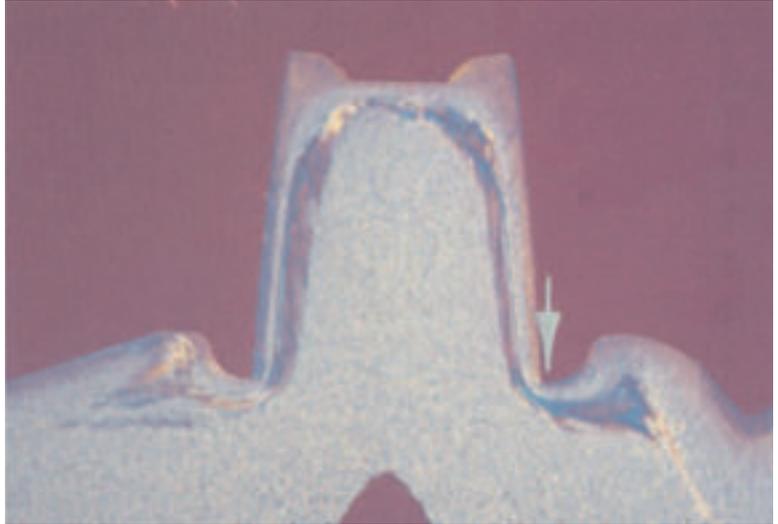
Part:
Bobbin

Resin:
PA 66

Problem:
Bobbin heads break in assembly.

Reason for Failure:
High shear at gate located at head of bobbin. In addition sharp corner at shoulder (see arrow).

Remark:
Sharp corners increase brittleness even with parts of conditioned nylon.



M: 25

A Shear at gate

Microtome Cut -

Thin Polished Section ×

Thickness 15 μm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz ×

Shear Orientation

Part:
Snap-hinge

Resin:
PA 66, modified, black

Problem:
Break in hinge after few flexions.

Reason for Failure:
Too high shear orientation due to too low melt temperature.

Remark:
Additional notch effect in corner of flexioned area caused by damaged surface of mould (see arrow in photo B).



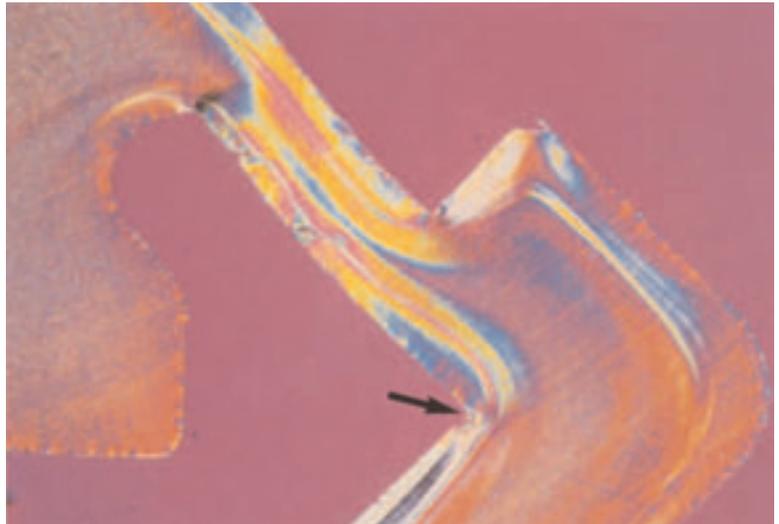
M: 13

A Shear and inhomogeneous melt

Microtome Cut	-
Thin Polished Section	×
Thickness	15 μm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	×
Pol. Filter + Red Quartz	×	×



M: 32

B Enlargement of A

Shear Orientation

Part:
Connector hinge

Resin:
PA 66, modified

Problem:
After one flexion when closing hinge, breakage occurred. High reject rate on assembly line.

Reason for Failure:
The shear orientation – inevitable in hinges – was increased due to too low or inhomogeneous melt temperature.

Remark:
Even without presence of shear orientation cold or inhomogeneous melt causes brittleness of parts.



M: 14

A Hinge

Microtome Cut	–
Thin Polished Section	×
Thickness	10 μm
Microscopic Light	
Photo	A
Transmittant	×
Pol. Filter	×
Pol. Filter + Red Quarz	×

Pigment Agglomeration

Part:
Window handles

Resin:
POM, coloured with masterbatch

Problem:
Part had been moulded from natural resin coloured by adding masterbatch. Parts showed coloured streaks on surface.

Reason for Failure:
Inhomogeneous melt or too low melt temperature prevented uniform distribution of the colour masterbatch.



A Part

Microtome Cut	×
Thin Polished Section	-
Thickness	20 µm
Microscopic Light	
Photo	A B
Transmittant	- ×
Pol. Filter	- ×
Pol. Filter + Red Quartz	- -



M: 25

B Cross section at area with streaks

Pigment Agglomeration

Part:
Cover

Resin:
POM, coloured with red masterbatch

Problem:
Spots of darker red visible on surface. Rejected by quality control.

Reason for Failure:
Microtome cut through area with dark spot revealed agglomeration of masterbatch. The high concentration of red batch caused darker spots of red on surface.

Remark:
Insufficiently dispersed masterbatch may indicate inhomogeneous melt.



M: 50

A Particle of masterbatch

Microtome Cut ×

Thin Polished Section –

Thickness 25 µm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz ×

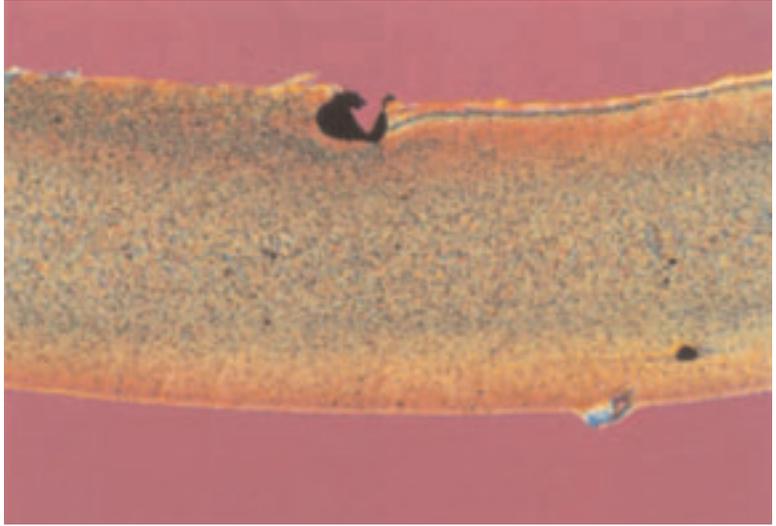
Pigment Agglomeration

Part:
Numbering wheel

Resin:
POM

Problem:
Material had been coloured black with dry pigment. Gears were brittle in use.

Reason for Failure:
Colouring with dry pigment caused pigment agglomeration which acts like foreign particles causing premature breakage.



M: 80

A Breakage area with pigment concentration

Microtome Cut ×

Thin Polished Section -

Thickness 20 µm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red. Quartz ×

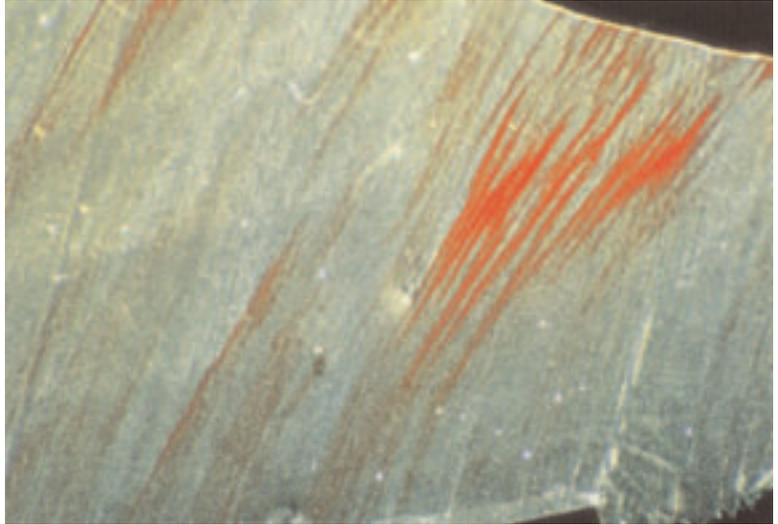
Pigment Agglomeration

Part:
Extruded rod

Resin:
PA 66, modified, coloured with masterbatch

Problem:
No uniform colour was obtained with red masterbatch. Red streaks on surface visible.

Reason for Failure:
Inhomogeneous melt prevented uniform dispersion of masterbatch.



M: 25

A Section with streaks

Microtome Cut ×

Thin Polished Section -

Thickness 25 µm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz -

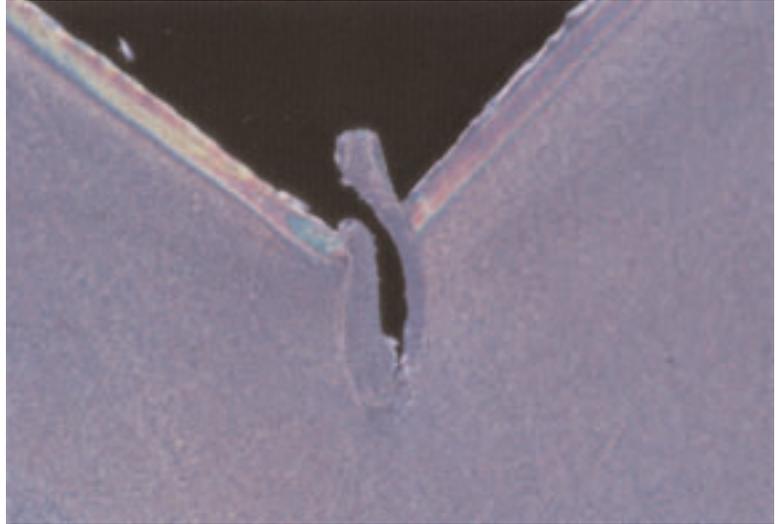
Notch Effect due to flashing/ sharp corners

Part:
Cam shaft gear

Resin:
POM

Problem:
Breakage of teeth.

Reason for Failure:
Flash in tooth base due to damaged mould caused notch effect resulting in premature breakage.



M: 65

A Notch due to flash

Microtome Cut	×
Thin Polished Section	-
Thickness	20 µm
Microscopic Light	
Photo	A
Transmittant	×
Pol. Filter	×
Pol. Filter + Red Quartz	-

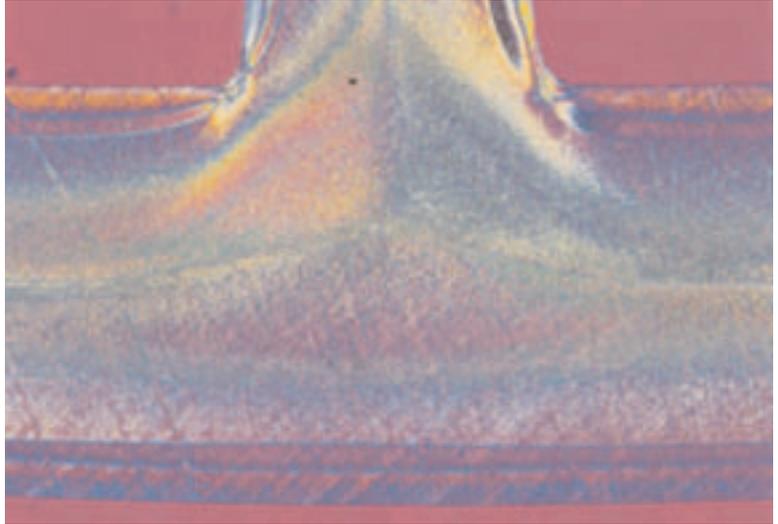
Notch Effect due to flashing/ sharp corners

Part:
Body with ribs

Resin:
POM

Problem:
Surface cracks at ribs in some parts of the delivered series.

Reason for Failure:
Cracks caused by notch effect from sharp corners in tool. High stress orientation at corners in comparison to non-failing parts.



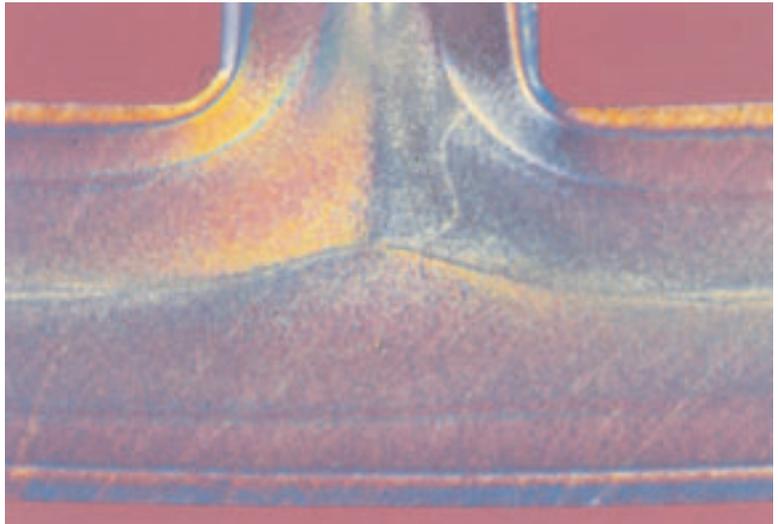
M: 50

A Stress orientation at sharp corners

Microtome Cut	–
Thin Polished Section	×
Thickness	15 µm

Microscopic Light

Photo	A	B
Transmittant	×	×
Pol. Filter	×	×
Pol. Filter + Red Quartz	×	×



M: 50

B Corners well rounded off

Notch Effect due to flashing

Part:
Ball bearing cage

Resin:
POM

Problem:
Trial to evaluate "overmoulding"
causing flashing on parts.

Reason for Failure:
Flash resulted in notch effect during
assembly.



A Part

Microtome Cut -

Thin Polished Section -

Thickness -

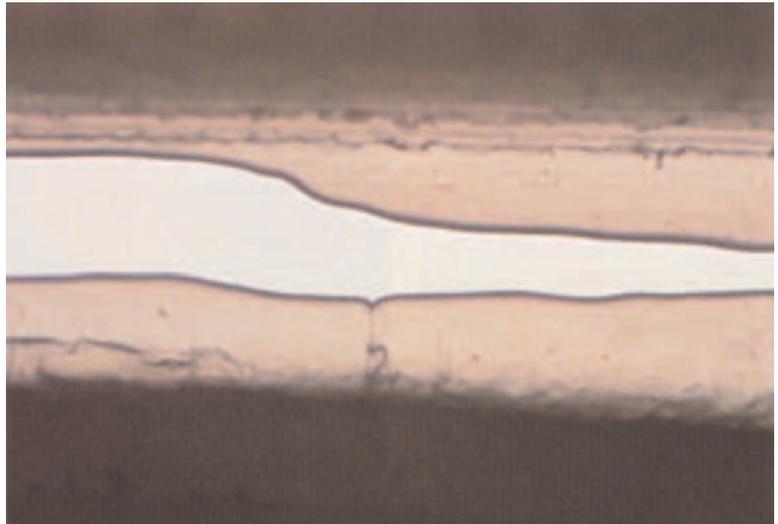
Microscopic Light

Photo A B

Transmittant - x

Pol. Filter - -

Pol. + - -
Red Quartz



M: 35

B Flash

Notch Effect due to flashing

Part:
Distance rings

Resin:
PA 66, dyed after moulding

Problem:
Rings crack when mounted on an axle.

Reason for Failure:
The thin flash cracked under load propagating a notch effect causing the breakage of the ring.

Remark:
Insufficient clamping force of moulding machine could be the reason for the pronounced flash.



A Part

Microtome Cut -

Thin Polished Section -

Thickness -

Microscopic Light

Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	-	-
Pol. Filter + Red Quartz	-	-	-



M: 28

B Flash



M: 28

C Broken flash causing notch effect

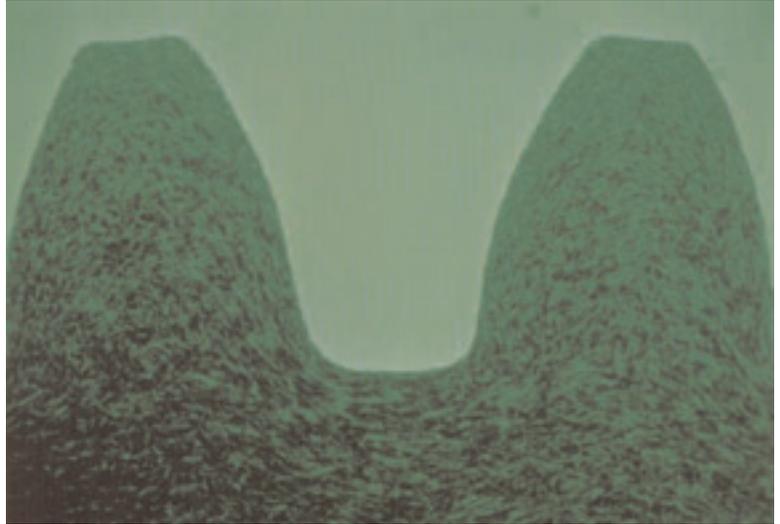
Glass fibre Orientation

Part:
Gear

Resin:
PA 66, 30% glass fibre content

Problem:
No reject. Study of glass fibre orientation in teeth of gear.

Reason for Failure:
Glass fibres follow the shape of the teeth well. No turbulences or glass fibre agglomeration can be seen.



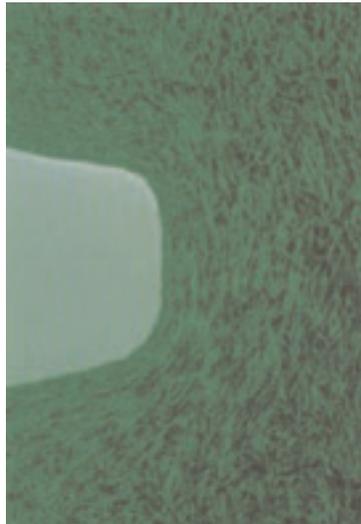
M: 12

A Teeth of gear

Microtome Cut	-
Thin Polished Section	×
Thickness	20 μm

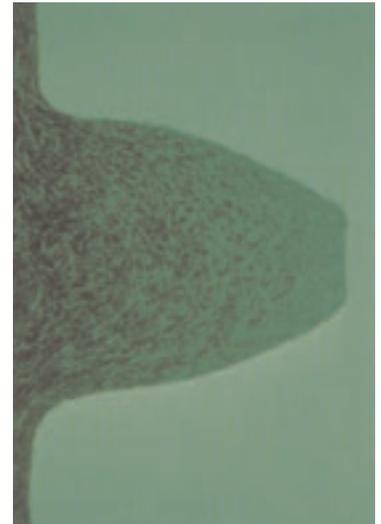
Microscopic Light

Photo	A	B	C
Transmittant	×	×	×
Pol. Filter	-	-	-
Pol. Filter + Red Quartz	-	-	-



M: 22

B Tooth



M: 17

C Between two teeth

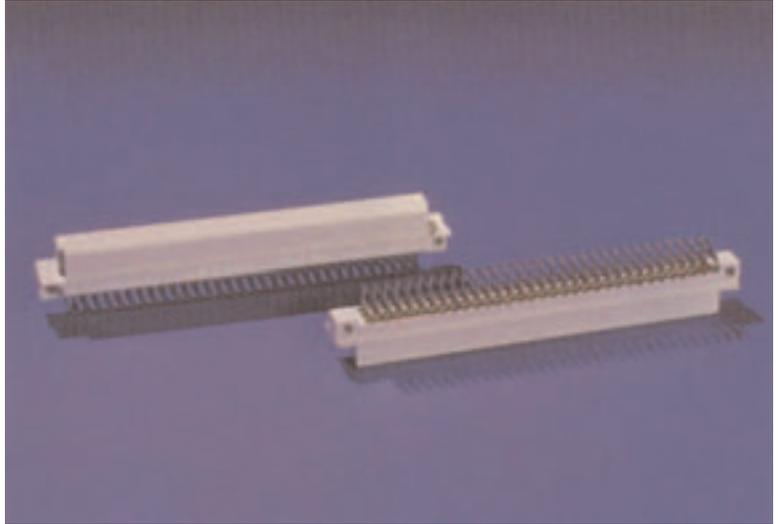
Glass fibre Orientation

Part:
Connector

Resin:
PETP, 30 % glass fibre content

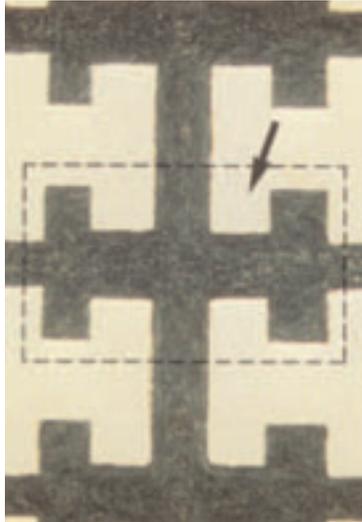
Problem:
Dimensional differences of holes within one connector.

Reason for Failure:
By recognizing existing glass fibre orientation, slight changes in moulding conditions reduced differences in dimension of holes within part.



A Parts

Microtome Cut	-		
Thin Polished Section	x		
Thickness	10 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	-	x	x
Pol. Filter	-	-	-
Pol. Filter + Red Quartz	-	-	-



M: 7



M: 22

B Fibre orientation

C Enlargement of B

Welding

Part:

Ball, spin welded
(part for demonstration)

Resin:

POM

Problem:

Two sphere halves are spin welded.

Remark:

Good weld giving resistance to high burst pressure.



M: 32

A Good spin weld

Microtome Cut ×

Thin Polished Section -

Thickness 25 μm

Microscopic Light

Photo A

Transmittant ×

Pol. Filter ×

Pol. Filter +
Red Quartz -

Welding

Part:
Several spin welded parts

Resin:
POM

Problem:
Part not gas-tight in welded area.

Reason for Failure:
Area of spin weld not optimally designed. Too sharp angle which cannot be filled and thus did not weld.



A Parts, spin welded

Microtome Cut	×		
Thin Polished Section	-		
Thickness	25 μm		
Microscopic Light			
Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	×	×
Pol. Filter + Red Quartz	-	-	-



M: 17

B Weld area not gas-tight



M: 17

C Good spin weld

Welding

Part:
Different vibration welded parts

Resin:
POM, PA 66

Problem:
Quality control of welded area.

Remark:
All welded areas are good.

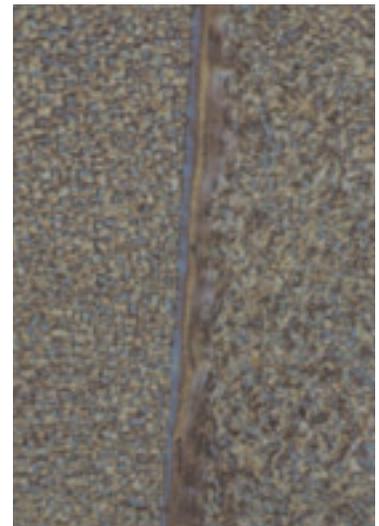


A Parts, vibration welded

Microtome Cut	×		
Thin Polished Section	-		
Thickness	25 µm		
Microscopic Light			
Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	×	×
Pol. Filter + Red Quartz	-	-	-



M: 22.5



M: 22.5

B Good weld – POM

C Good weld – PA 66/PA 66 GR

Welding

Part:
Fuel filter, spin welded

Resin:
PA 66

Problem:
Weld has sufficient mechanical strength but some are not gas-tight.

Reason for Failure:
Poor quality of weld preventing good bond between the two halves.

Remark:
Parts which are considered to be of sufficient mechanical strength do not have a perfect weld either. Furthermore, melt temperature of the moulded parts has been too low and inhomogeneous.



A Part, spin welded

Microtome Cut	×
Thin Polished Section	-
Thickness	25 µm
Microscopic Light	

Photo	A	B	C
Transmittant	-	×	×
Pol. Filter	-	×	×
Pol. Filter + Red Quartz	-	×	×



M: 35



M: 35

B Not gas-tight but mechanically acceptable

C Not gas-tight and insufficient mechanical strength

Welding

Part:

Box, ultrasonically welded
(part for demonstration)

Resin:

POM, PA 66

Problem:

POM = insufficient weld strength
PA 66 = no welding effect

Reason for Failure:

- POM
- photo B: too long weld time
- photo C: bad dimensional fitting
- photo D: good weld
- photo E: good weld, different part
PA 66
- photo F: material contains too much
moisture
- photo G: good weld, dry material



A Part

Microtome Cut	×
Thin Polished Section	-
Thickness	20 µm

Microscopic Light

Photo	A	B to G
Transmittant	-	×
Pol. Filter	-	×
Pol. Filter + Red Quartz	-	×



M: 22

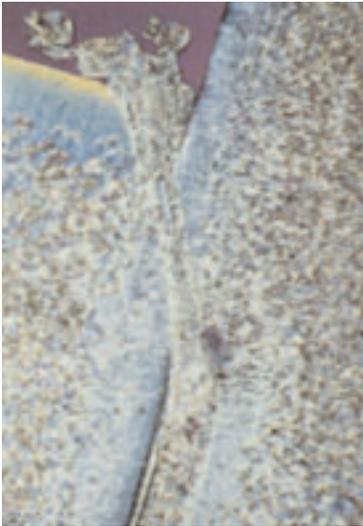


M: 22

B Weld time too long – POM

C Bad fitting – POM

Welding



M: 22

D

Good weld – POM



M: 17

E

Good weld – POM, different part



M: 22

F

Material moist – PA 66



M: 22

G

Good weld – PA 66

We believe this information is the best currently available on the subject. It is offered as a possible helpful suggestion in experimentation you may care to undertake along these lines. It is subject to revision as additional knowledge and experience are gained. Du Pont makes no guarantee of results and assumes no obligation or liability whatsoever in connection with this information. This publication is not a licence to operate under, or intended to suggest infringement of any existing patents.

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