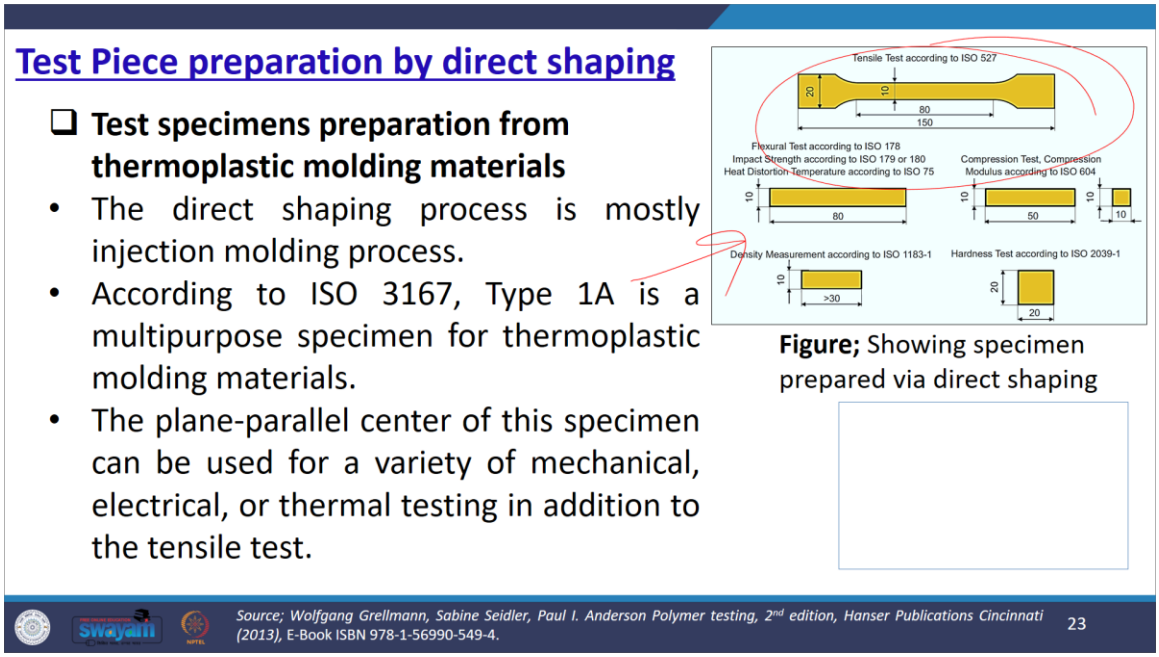
Lecture 37 - Polymer testing-02 (Standardization, Sample preparation)

Standardization plays a crucial role in ensuring consistency and reliability in product quality and testing procedures. Standards are documents that outline specifications for products, services, or testing methods, providing a common language and framework for various industries. They guide the manufacturing, testing, and usage of materials, ensuring compatibility and safety.

In the field of polymer testing, standards help establish protocols for testing different properties of polymers. This includes methods for measuring mechanical, thermal, chemical, and other characteristics. Adhering to standardized testing methods ensures that results are comparable across different laboratories and testing facilities.

Sample preparation is a key step in the testing process. It involves creating specimens that accurately represent the material being tested. There are two main approaches to sample preparation: direct shaping and indirect shaping.

1. **Direct Shaping:** Direct shaping involves forming the polymer material directly into the desired shape for testing. This can include processes like injection molding, compression molding, or extrusion. The goal is to create specimens that closely resemble the final product or component.



Figure; Showing specimen prepared via direct shaping

2. Indirect Shaping: Indirect shaping involves creating specimens through cutting, machining, or otherwise modifying existing materials. This approach is useful when direct shaping is not practical or when testing requires specific geometries or dimensions.

Both direct and indirect shaping methods aim to produce samples that are representative of the material's properties in real-world applications. The choice between these methods depends on factors such as material characteristics, testing requirements, and equipment availability.

Standardization and proper sample preparation are integral to the field of polymer testing. Standards provide a framework for consistent and reliable testing, while effective sample preparation ensures that the tested specimens accurately reflect the material's properties. These practices contribute to the overall quality, safety, and performance of polymer-based products in various industries.

Standards and standardization are crucial components that contribute significantly to the success of the economy and society. They provide a common framework for comparing and ensuring the quality and reliability of products and testing procedures. Deviations from established standards are indicators of potential failures, prompting the need for addressing and rectifying issues.

Government regulations become less necessary in the presence of effective standards and standardization. Technical convergence is facilitated through standard committees, such as the Standard Committee on Material Testing of the German Institute of Industry Standard (DIN) organization, which develops new approaches to meet evolving requirements. In the Indian context, the Bureau of Indian Standards (BIS) plays a similar role, covering a wide range of standards relevant to the Indian context.

Standards for conducting tests and specifying test apparatus and specimens are established to guarantee the repeatability and reproducibility of material testing techniques, particularly in polymer testing. The proper specification of processes, specimens, and test apparatus is crucial for maintaining consistency and reliability in testing procedures.

Sources of standards can be categorized into three groups: international organizations like the International Organization for Standardization (ISO), national organizations like BIS, and individual companies that may have their own internal standards.

Several factors influence the setting of national and international standards, including globalization, market expansion into new economic zones, the demand for shorter development times, and the increasingly shorter life service cycles of products. The resulting standards promote efficiency and quality assurance across various sectors, including business, technology, science, and administration, aligning with fundamental principles for producing standards.

Standards serve a dual purpose. Firstly, they play a crucial role in ensuring the safety of people, machinery, technologies, and processes. Secondly, they serve as a means of

driving improvement in quality across various aspects of life and business. Standards make it simple to compare product attributes or manufacturing processes when applied. The ability of every member of the standard community to meet the technical and scientific requirements laid out in standards forms the basis for all kinds of comparisons, such as DIN, EN, ISO, or DIN.

Standards like DIN, EN, and ISO, among others, achieve the status of national standards through the harmonization of international and European standards. International standards like ISO, European standards (EN), and Indian standards (BIS) are widely recognized and used. The American ASTM standards from the American Society for Testing Materials are also commonly employed on a global scale. These standards encompass test methods and procedures for testing polymers and composites, providing a platform for the development and dissemination of standards and test procedures on a global scale.

ASTM International standards play a vital role in product testing, research and development, and quality control. ASTM has a dedicated committee focused on plastics, responsible for generating over 500 test procedures, recommendations, practices, and guidelines. This committee is involved in ongoing evaluation, updating existing standards, and creating new protocols.

It's important to distinguish between ASTM and ISO standards, particularly concerning specimen geometry, size, and test condition requirements. ISO/IEC standards, taking on the role of European standards, specify parameters for evaluating organizational structure, the use of test staff, technical facilities, the creation of test reports, testing and calibration work methods, and the development of measuring uncertainty. These standards emphasize the use of test results in quality assurance and design.

Testing facilities with their own test standards, based on extensive experience, have their competency recognized in various areas covered by standards. National regulations related to the responsibility for defective products provide a crucial legal foundation for material testing. Product liability laws outline necessary measures for material and polymer testing, emphasizing the selection of suitable and instructive test procedures, and ensuring that design and assembly are easily testable.

Testing must be conducted using accepted techniques that produce meaningful results. Test results should be assessed in light of their intended purpose, which includes property, product, and process observations, proactive mistake prevention, and failure analysis as needed. Various standards contribute to the fundamental aspects of standardization, reporting, and quality management systems. Examples of relevant ISO and ASTM standards include:

1. **DIN 8201:1994** - Standardization fundamentals.

2. **ISO 9000** - Quality management system fundamentals and vocabulary.
3. **ISO 9001** - Quality management system requirements.
4. **ISO 9004** - Guidelines for performance improvement in quality management systems.
5. **ISO 17025** - General requirements for the competence of testing and calibration laboratories.
6. **ISO 291** - Standard atmosphere for conditioning and testing of plastics.
7. **ASTM standards** - Examples include practices for conditioning plastics, testing plasticizers, preparation of flat composite panels, and sampling rubber testing.

Test specimen preparation is crucial and should adhere to the standards being followed, such as BIS, ISO, or ASTM. The specimen's dimensions and condition must meet the requirements specified in the applicable standards. This precision is essential for accurately characterizing the mechanical, thermal, or electrical properties of polymer molding materials.

The creation of test specimens is a critical step in examining the properties of polymer materials. These specimens can be produced separately or extracted from components or plastic parts for profiling, failure studies, or other analyses. The methods of creating these specimens fall into two categories: direct shaping processes and indirect shaping processes.

Direct Shaping Processes:

1. **Injection Molding:** Utilizes the injection of molten material into a mold cavity.
2. **Injection Stamping:** Involves the stamping of material in an injection process.
3. **Compression Molding:** Applies pressure to shape the material in a mold.
4. **Casting:** Forms materials by pouring them into a mold.

Indirect Shaping Processes:

1. **Extrusion:** Produces shapes by forcing material through a die.
2. **Calendaring:** Shapes material between rollers.
3. **Stamping:** Forms material by stamping it.
4. **Cutting:** Creates shapes by cutting the material.

During the shaping process, various phenomena occur, such as shearing during injection, stretching and orienting of macromolecules, and the cooling and curing sequence. These phenomena cause deformations and significantly influence the internal state of the component or specimen.

Substances may experience process shrinkage, also known as specific volume shrinkage, as they transition from a liquid to a solid form. To account for this, molds are designed with a comparable oversize. Shrinkage can impact dimensional stability and tolerance, with filled or reinforced materials typically experiencing less shrinkage than the matrix material. Understanding these processes is crucial for designing molds and ensuring the accuracy of test specimens.

Continuing with the discussion on the preparation of test specimens by direct shaping, particularly for thermoplastic molding materials, the focus is on the injection molding process. According to ISO standards, a type 1A specimen is considered a multipurpose specimen suitable for various mechanical, electrical, or thermal tests, including tensile tests.

The advantages of using type 1A specimens include providing a uniform reference standard for orientation and residual stress, along with identical thickness and width. However, in some cases, it may be challenging to extract a 170 mm long specimen from molded parts or components. In such situations, proportionally scaled-down specimens can be prepared, but it's crucial to adjust the test speed and strain measurement accordingly.

Two primary approaches are outlined to minimize the effects of processing conditions or establish a clear reference standard:

1. **Initial State Specimen Preparation:**
    - The initial state specimen should be macroscopically isotropic, lacking preferred orientation.
    - It should be uniform concerning the distribution of morphological texture and free from residual stresses.
    - Compression molding is one method to produce this initial stage, creating conditions that may not exist in real components or molded parts.
    - Specimens can be cut from plates or directly formed in a compression mold.
2. **Preparation of Samples with Processing-Dependent Reference State:**

- This involves preparing specimens with a reference state that depends on the processing conditions.
- Preheating, limited shear effects during compression, and controlled slow cooling rates can help minimize the creation of residual stress and orientation.

These approaches aim to ensure that the test specimens accurately represent the intended material properties without the influence of undesirable factors introduced during the molding process.

In compression molding, it is essential to carefully choose the pressure and temperature parameters. The degree of crystallinity and the structure, either crystallite or spheroidal, are controlled by the cooling rate in semi-crystalline polymers. Practical experience suggests that, for achieving sufficient homogeneity, the holding temperature should be approximately 100 degrees Celsius higher than the softening temperature of the amorphous or semi-crystalline molding material.

The provided example outlines the compression molding specifications for various plates and specimens made from polystyrene molding materials (PSN, ABS). The specifications include holding temperature, holding pressure, preheat time, and holding time. These conditions are designed to produce homogeneous plates or specimens that are free of tension and orientation.

For creating multipurpose specimens following ISO standards and achieving a reference state, injection molding is the preferred method. Manufacturers optimize the specifications for molding materials based on injection molding, considering parameters such as pressure, duration, temperature, machine type, and mold architecture.

The internal state resulting from chosen processing and mold parameters, pressure duration, temperature, machine type, and mold architecture (including flow distance and measurement) plays a crucial role in determining the specimen's condition. Dimensional stability and precision of plastic products are significantly influenced by the material's capacity to shrink at higher temperatures and mold cooling contraction. Proper mold design and draft angle help mitigate contraction resulting from volume contraction during the transition from a liquid to a solid state in the processing technology.

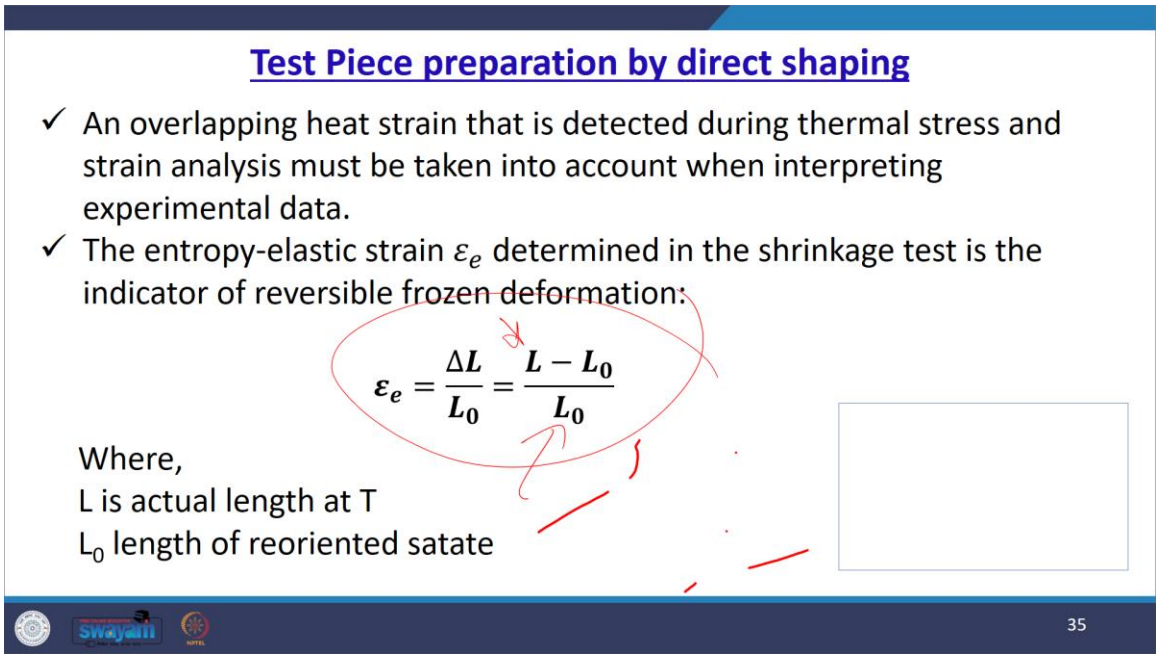
The shrinkage of a component or specimen is a result of molecular orientation relaxation during heating, leading to changes in macroscopic dimensions and length. This change creates a state that is entropically unfavorable relative to the original state. Various effects contribute to this change:

1. **Plastic deformation:** Occurs during processes like deep drawing or extrusion.

2. **Thickness differences:** Lead to residual stresses and flow lines during injection molding.
3. **Orientation:** Arises during extrusion and injection molding, resulting in anisotropic properties.
4. **Surface texture and rough spots:** Along with notches, stresses, and static weld lines.

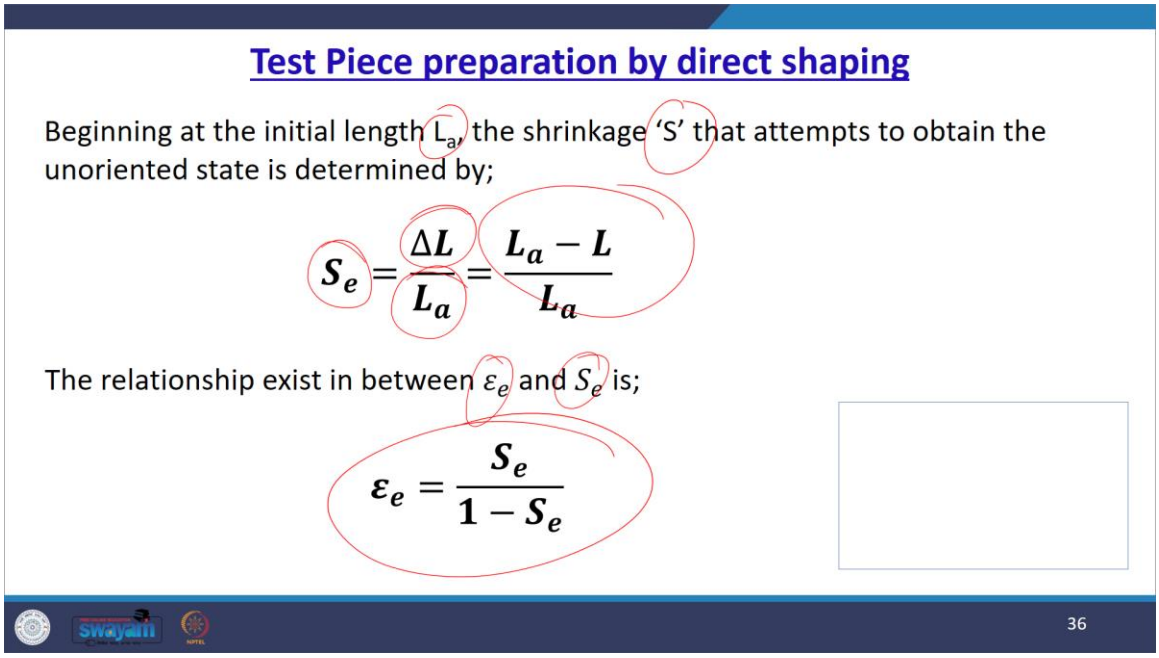
When temperature is raised and recovery is not prevented, shrinkage is observed in mechanically shaped, stretched, or injection-molded parts due to altered physical structure. The memory effect, or thermally induced reorientation processes, is the root cause of these effects as it alters the entropy state.

Free shrinkage is often characterized as a particular kind of retardation, while controlled shrinkage is additionally characterized as residual stress relaxation. Overlapping heat strain detected during thermal stress and strain analysis should be considered when interpreting experimental data.



The entropy elastic strain determined in the shrinkage test serves as an indicator of reversible frozen deformation, given by the equation: $\varepsilon_e = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0}$, where ΔL is the change in length, L is the actual length at temperature T, and L_o is the reoriented state. If the initial length is denoted as L_a and the shrinkage (S_e) attempting to obtain the unoriented state is given by $S_e = \Delta L / L_a$, then there exists a relationship between ε_e and S_e:

$$\varepsilon_e = \frac{S_e}{(1 - S_e)}$$



When shrinkage is prevented, the measured shrinkage force F_s can be represented as the shrinkage stress σ_s, where $\sigma_s = \frac{F_s}{A_0}$, and $\sigma_s = \frac{\sigma_s(T_g)T}{T_g}$ where:

- *Fs* is the shrinkage force.
- A_0 is the initial cross-sectional area of the specimen.
- *T* is the actual temperature.
- T_g is the glass transition temperature.
- $\sigma_s(T_g)$ is the shrinkage stress frozen at the glass transition temperature.

Test Piece preparation by direct shaping

When shrinkage is prevented, the measured shrinking force Fs can be represented as a shrinkage stress (σ_s)

$$\sigma_s = \frac{F_s}{A_0} \qquad \Rightarrow \sigma_s = \sigma_s(T_g)\frac{T}{T_g}$$

Where,

A_0 is initial cross section area of specimen

T is actual temperature

Tg is glass transition temperature

$\sigma_s(T_g)$ is the shrinkage stress frozen at glass transition temperature

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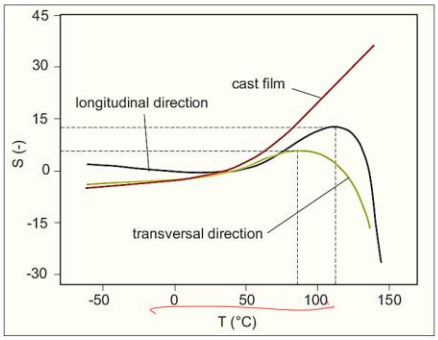
Performing a shrinkage test involves measuring the shrinkage force under specific conditions. In a practical example shown in a figure, a shrinkage test was performed on a biaxially stretched 30 µm thick polypropylene film under constant warming conditions (2 °C/min). At a noticeable high temperature, the film, mechanically loaded in the primary orientation direction, starts to shrink.

The figure compares the shrinkage of the biaxially stretched polypropylene film in the direction of orientation and transverse orientation with an unoriented PP cast film. This behavior is influenced by the greater amount of secondary valence bonding and stronger stretching of molecules in the major orientation direction compared to the cross-sectional direction.

Test Piece preparation by direct shaping

Shrinkage test



Figure; Demonstrates the results of a shrinkage test performed on a biaxially stretched, 30 µm thick PP film under constant warming conditions of 2 °C/min. At noticeably higher temperatures, the film that has been mechanically loaded in the primary orientation direction starts to shrink.

Figure; shrinkage of biaxial stretched Type equation here.PP film in the direction of orientation and transvers orientation compared with an unoriented PP cast film

Source; Wolfgang Grellmann, Sabine Seidler, Paul I. Anderson Polymer testing, 2nd edition, Hanser Publications Cincinnati (2013), E-Book ISBN 978-1-56990-549-4. 38

As the temperature rises, a cast film created without orientation continually lengthens, displaying linear thermal expansion behavior. The figure represents the shrinkage force *Fs* dependent on the temperature at a constant 0.1% strain for biaxially stretched polypropylene film and polypropylene cast film. The shrinkage versus temperature diagram for the film under investigation is illustrated.

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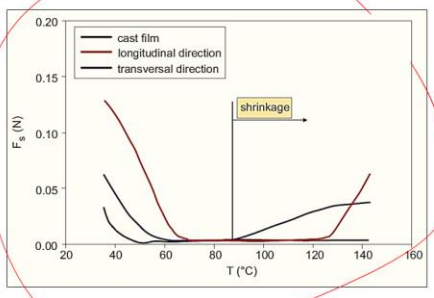


- For the film under investigation, shrinkage vs. temperature diagrams are displayed.
- The evolution of the curve and the temperature levels at transition show the variable anisotropy (orientation) of the specimens produced by their various processing settings.

Figure; Shrinkage Force Fs dependent on temperature at constant 0.1% strain on biaxial stretched PP film and PP cast film

Source; Wolfgang Grellmann, Sabine Seidler, Paul I. Anderson Polymer testing, 2nd edition, Hanser Publications Cincinnati (2013), E-Book ISBN 978-1-56990-549-4. 40

The curve's evolution and the temperature level at the transition demonstrate the variable anisotropic orientation of the specimen produced by various processing settings. Depending on the degree of direction, the observed force decreases with rising

temperature. According to Hooke's law, this decline stems from a decrease in Young's modulus at higher test temperatures, and shrinkage begins around 90 degrees Celsius in the direction of orientation and 120 °C transverse to orientation.

The shrinkage test highlights the variation in shape stability and the tendency for shape distortion caused by elevated temperature. It is sensitive to the production process deformation kinetics. Conclusions regarding the state of molecular network transformation processes and technically significant limiting temperatures can be drawn from both tests.

Now, let's talk about specimens prepared by indirect shaping. Indirect shaping refers to the process of creating specimens by cutting them from larger finished plates that have been compression, extrusion, or injection molded.

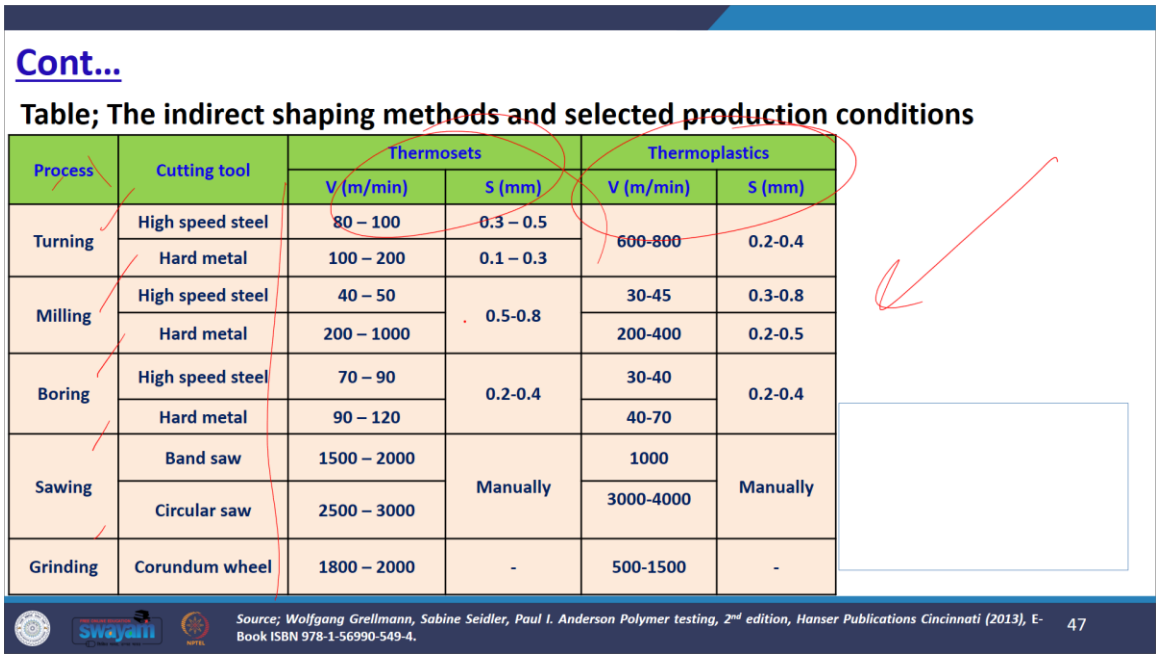
0 times the largest particle diameter of the molding material or a 5 millimeter 1 over 5 of the largest cross section of the specimen must always be maintained. Now, if the molding material contains a larger particle and a group of particles a homogeneous distribution of the particles, the test specimen must be selected such that the longest dimension of the standardized specimen is smaller than twice the largest particle diameter. The particle diameter must also be determined with regard to the shrinkage ratio, the orientation ratio and the internal state after processing and using a microscope. So, here we need to understand the particle diameter which is always taken as an upper limit and the definition for the particle is that any agglomerate or cluster is also considered as a single particle in this context. Now, if the measurement is close to the limit the results should only be evaluated if it is possible to draw a definite conclusion and the physical state after processing and the particle size need to be considered in order to interpret the measurement results. Now, here we need to consider the particle diameter in order to understand how the internal state is there and how it is impacting the performance. If the measurements are significantly under the limit then the test is representative only if the necessary statistical criteria are fulfilled and there is no clear departure from a normal distribution. Now, the particles must not be left in the specimen if it is possible to remove them and if the state of a specimen is dependent on the internal state after processing then the maximum measurement uncertainty must be allowed for the measurement. The selection of the specimen for a particle test must be done with regard to the diameter of the particles, the smallest size and the most likely form of occurrence and the test results should be reported in the test report. If the process is used then the process parameters must be documented and the estimation of the influence of the residual internal stress and the isotropic and isotropy and the thickness must be taken into account. Now, let us talk about how the indirect shaping is done and it is the injection molding process.

The details provided emphasize the importance of precision and careful consideration in the specimen preparation process, especially when using indirect shaping methods like

turning, milling, boring, sawing, and grinding. The note about maintaining a minimum thickness of 1.5 mm and using the shrinkage test for identifying process-related anisotropy adds practical insights into the testing procedures.

Furthermore, the mention of considerations such as the impact of milling tool diameter on lateral edges' quality, the need for adherence to specific rules for saw blades and milling tools, and the importance of maintaining parallelism during subsequent grinding or polishing operations underscores the meticulous nature of specimen preparation.

The table enumerating various indirect shaping methods, production conditions, and cutting tools for thermoset and thermoplastic materials provides a comprehensive overview, aiding researchers and practitioners in selecting appropriate techniques based on the material characteristics.



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Table; The indirect shaping methods and selected production conditions

Process	Cutting tool	Thermosets		Thermoplastics	
		V (m/min)	S (mm)	V (m/min)	S (mm)
Turning	High speed steel	80 – 100	0.3 – 0.5	600-800	0.2-0.4
	Hard metal	100 – 200	0.1 – 0.3		
Milling	High speed steel	40 – 50	0.5-0.8	30-45	0.3-0.8
	Hard metal	200 – 1000		200-400	0.2-0.5
Boring	High speed steel	70 – 90	0.2-0.4	30-40	0.2-0.4
	Hard metal	90 – 120		40-70	
Sawing	Band saw	1500 – 2000	Manually	1000	Manually
	Circular saw	2500 – 3000		3000-4000	
Grinding	Corundum wheel	1800 – 2000	-	500-1500	-

Source; Wolfgang Grellmann, Sabine Seidler, Paul I. Anderson Polymer testing, 2nd edition, Hanser Publications Cincinnati (2013), E-Book ISBN 978-1-56990-549-4.

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If you have any specific questions or if there's more information you'd like to explore, feel free to let me know!

Thank you for providing a comprehensive overview of the considerations involved in obtaining optimum results during machining processes for specimen preparation. The details about producing cold and smooth chips, maintaining low cutting speeds with supplementary cooling, and avoiding stress during cooling offer valuable insights into achieving high-quality results.

The emphasis on low feed rates for superior surface quality, along with the importance of ensuring the specimen's longitudinal axis is parallel to subsequent grinding or polishing

operations, highlights the meticulous approach required in the entire testing and specimen preparation process.

I'm glad we could explore various aspects of testing protocols and specimen preparation. If you or anyone else has further questions or if there's anything specific you'd like to discuss, please feel free to let me know. Additionally, if you have any specific references you'd like to share, feel free to provide them.