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Study of the effect of ASTM and ISO testing conditions on mechanical properties of polypropylene

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W. R. Grace

Outline

- Introduction to W. R. GRACE
- Overview of ISO and ASTM Standards
- Specimen Preparation Comparison
  - Specimen Dimension
  - Injection Molding Condition
- Mechanical Properties Comparison
  - Flexural Modulus
  - Tensile Property
  - Notched Izod Impact Strength
- Summary

A Global Leader

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Built on **talent, technology, and trust**, Grace is a **leading global supplier of specialty chemicals**.

Our two industry-leading business segments—Catalysts Technologies and Materials Technologies—provide innovative products, technologies, and services that enhance the products and processes of our customers around the world.

Global Public Company

- 3,900 employees in 30 countries
- Customers in 60 countries
- New York Stock Exchange (GRA)
- Holding more than 800 active U.S. patents
- 24 acquisitions since 2003
- Headquarters: Columbia, Maryland USA
- Founded in 1854

A $2 Billion Technology Leader

Refining Technologies

- 61% FCC
- 39% Hydroprocessing (ART®)

[1] Includes $447M in ART joint venture sales.

Specialty Catalysts

- 45% Polypropylene Catalysts
- 41% Polyethylene Catalysts
- 9% Chemical Catalysts
- 5% Process Licensing

Materials Technologies

- 37% Chemical Process
- 34% Coatings
- 29% Consumer/Pharma

~$2B
2017 Sales[2, 3]

~$5B
Market Cap[1]

80%
#1 or #2
Business Positions

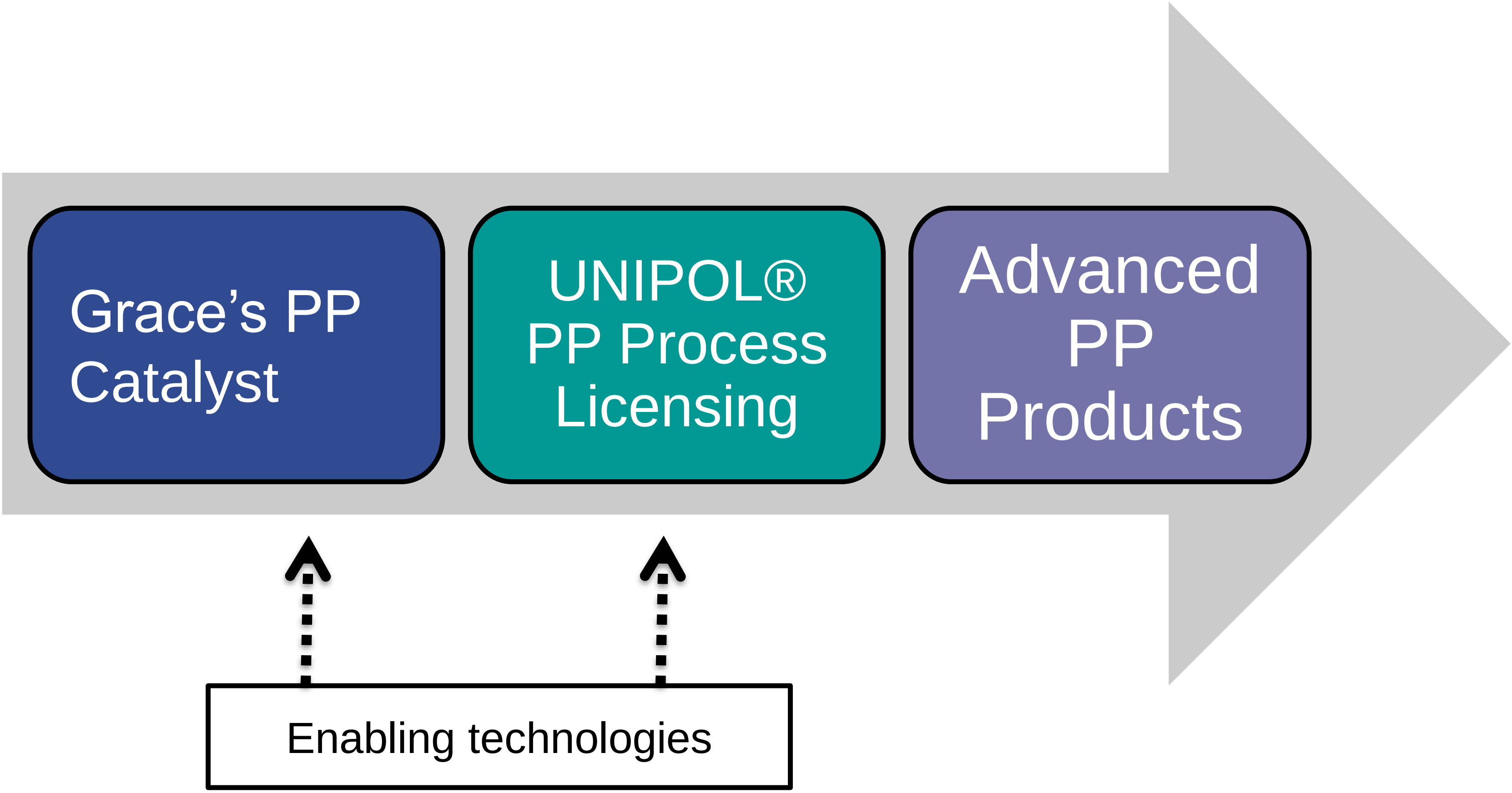
75%
Business Outside the US

#1

- FCC Catalysts
- Hydroprocessing
- Hydrocracking
- Polyolefin catalysts
- Independent polypropylene process technology licensing
- Specialty silica gel

[2] Based on FY2017
[3] Catalysts Technologies includes unconsolidated ART joint venture; FCC = Fluid Catalytic Cracking, SC = Specialty Catalysts, ART = Advanced Refining Technologies; FCC and ART together constitute the Refining Technologies operating segment

GRACE Licensing Innovation Strategy

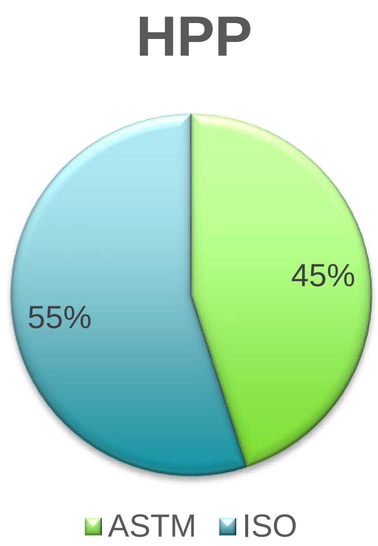


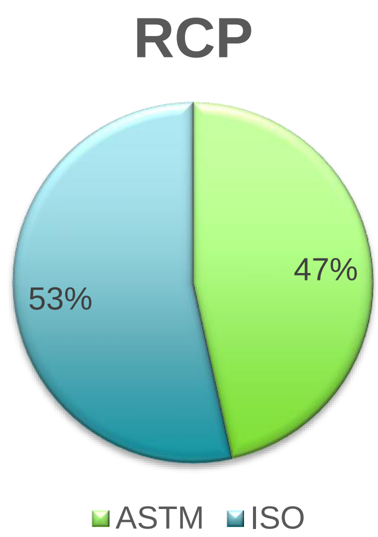
<u>Innovation strategy</u>: Drive improvements in PP product through catalyst and donor.

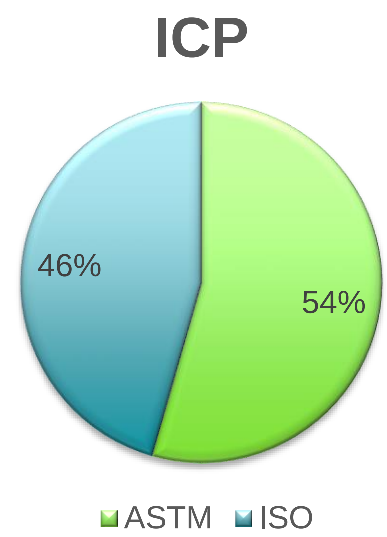
Overview of ASTM and ISO Standard

- ASTM and ISO are accepted globally
- Preference in different regions and countries

UL Prospector Statistics







The ASTM and ISO are equivalently applied in the Data Sheet

Overview of ASTM and ISO Standard

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	ASTM method	ISO method
Injection Molding	D4101,D3641	1873, 294
Flexural Properties	D790	178
Tensile Properties	D638	527
Notched Izod Impact Resistance	D256	180

Specimen Preparation Comparison: Dimensions

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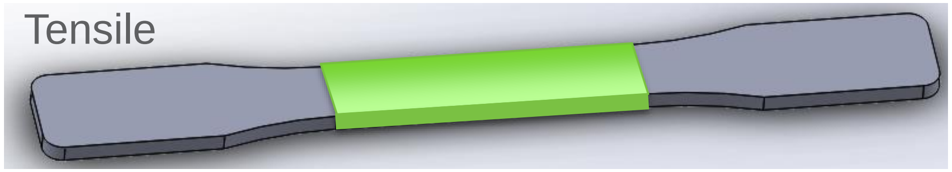
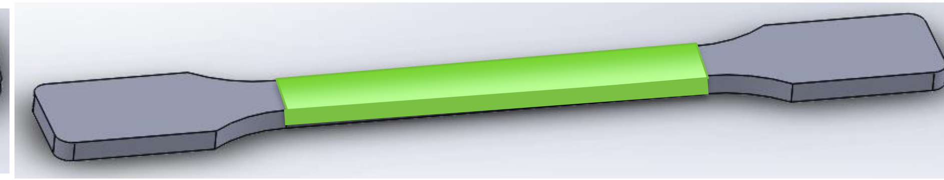
ASTM

ISO

Tensile





Narrow Section: 57 x 12.7 x 3.2 mm

Narrow Section: 80 x 10 x 4 mm

Flex

127 x 12.7 x 3.2 mm

80 x 10 x 4 mm

IZOD

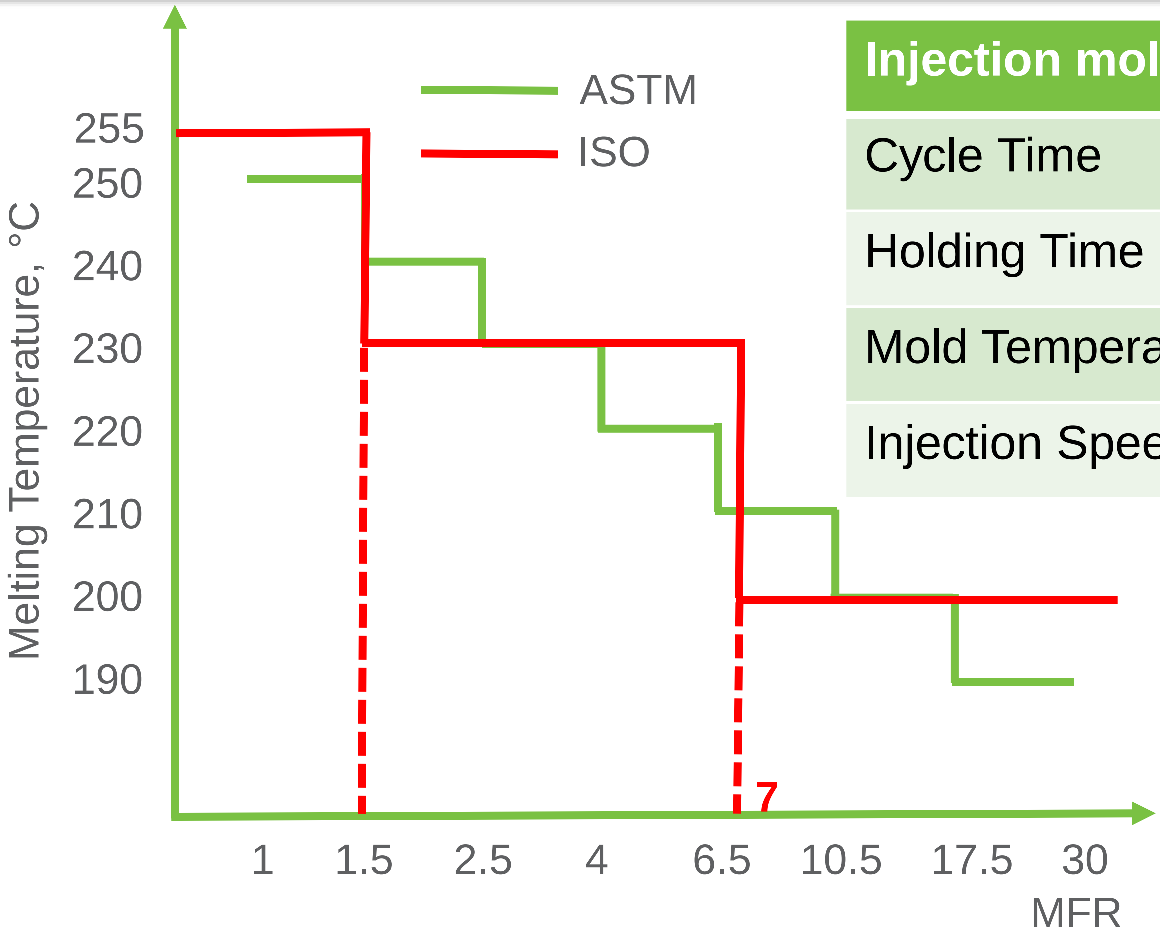
63.5 x 12.7 x 3.2 mm

80 x 10 x 4 mm

Cross Section: 40.6 mm^2 vs. 40 mm^2

- ISO specimen is narrower but thicker than ASTM specimen
- Similar cross section for ISO and ASTM specimen

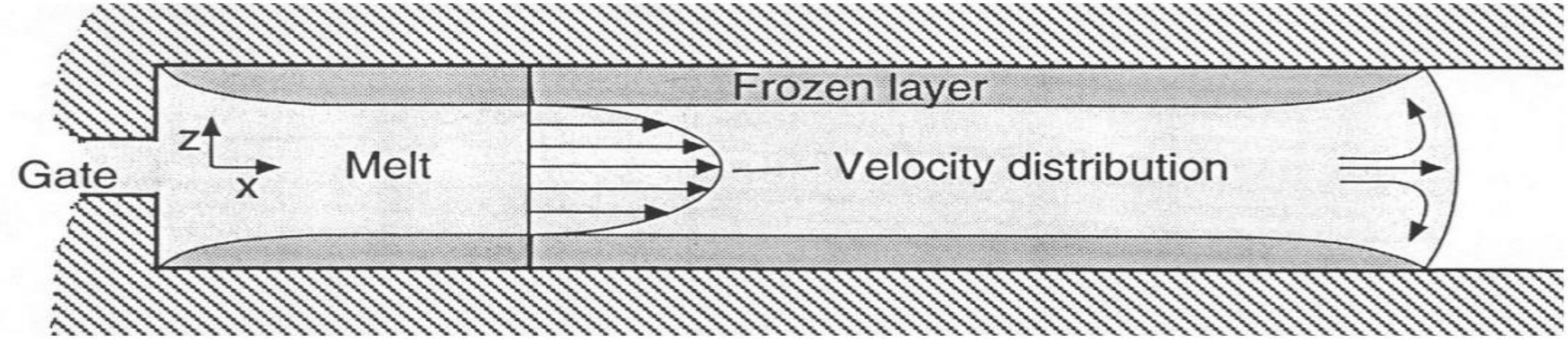
Specimen Preparation Comparison: IM Conditions

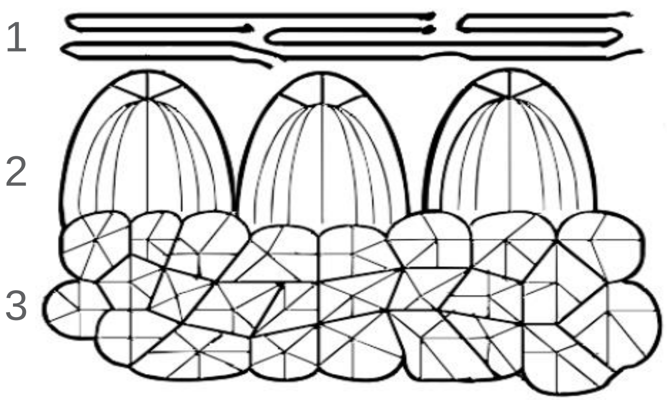


Injection molding	ASTM	ISO
Cycle Time	45 s	60 s
Holding Time	15 s	40 s
Mold Temperature	60°C	40°C
Injection Speed	200 mm/s	200 mm/s

- Melt temperature are the same only for MFR 2.5-4 and 10.5-17.5
- Injection molding ISO conditions use lower mold temperature and longer cycle time

Molding conditions affect part morphology





- Morphological structure of injection molding part:

1. Non-spherulitic skin
2. Intermediate zone
3. Spherulitic core

- Skin layer and intermediate zone morphology dominate the properties

Woodward, A. E. Understanding Polymer Morphology; Hanser Publishers; Munich, 1995

Injection Molding using Moldex 3D

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	ASTM	ISO
Melt Temperature	230C	230
Mold Temperature	60	40
Max. Inj. Pressure, MPa	50 MPa	50 MPa
Packing Time	15	40
Cooling Time	20	13
Mold Opening Time	5	5
Injection Speed, cm^3/s	40	40

Material Parameters	
MFR, g/10min @230°C	2.5
Thermal Conductivity	1.5E-3 J/sec.com.°C
Heat Capacity	3.1 J/g. °C
Viscoelasticity	White-Metzner Model
Crystallization	Appendix I
Modulus relaxation	Appendix II
PVT	Appendix III
Viscosity	Appendix IV

Temperature Distribution

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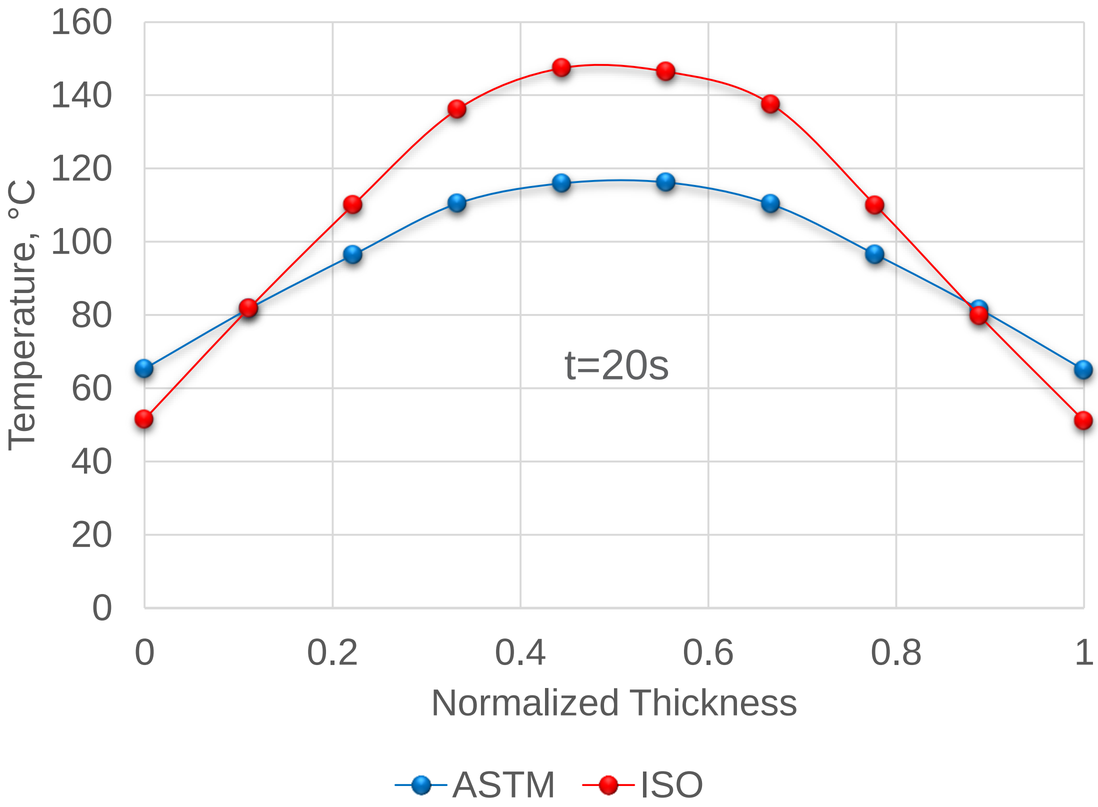
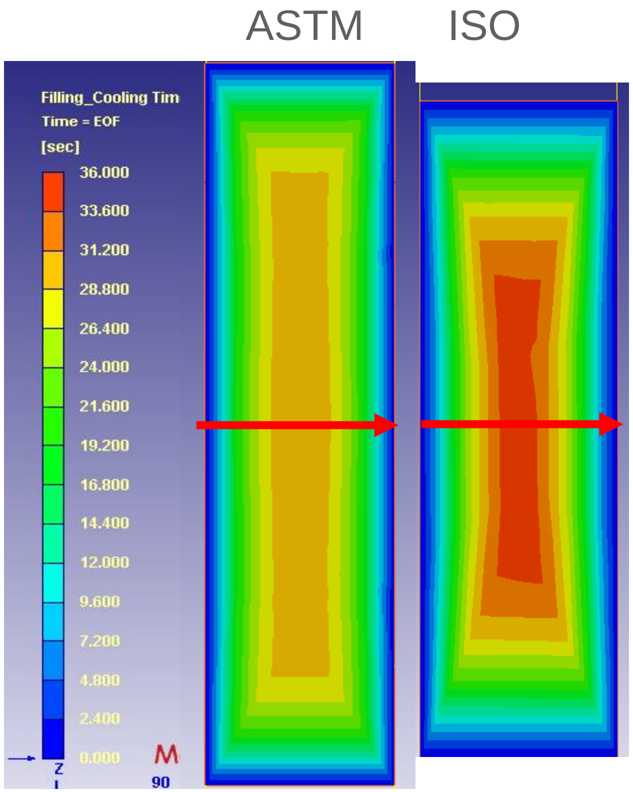
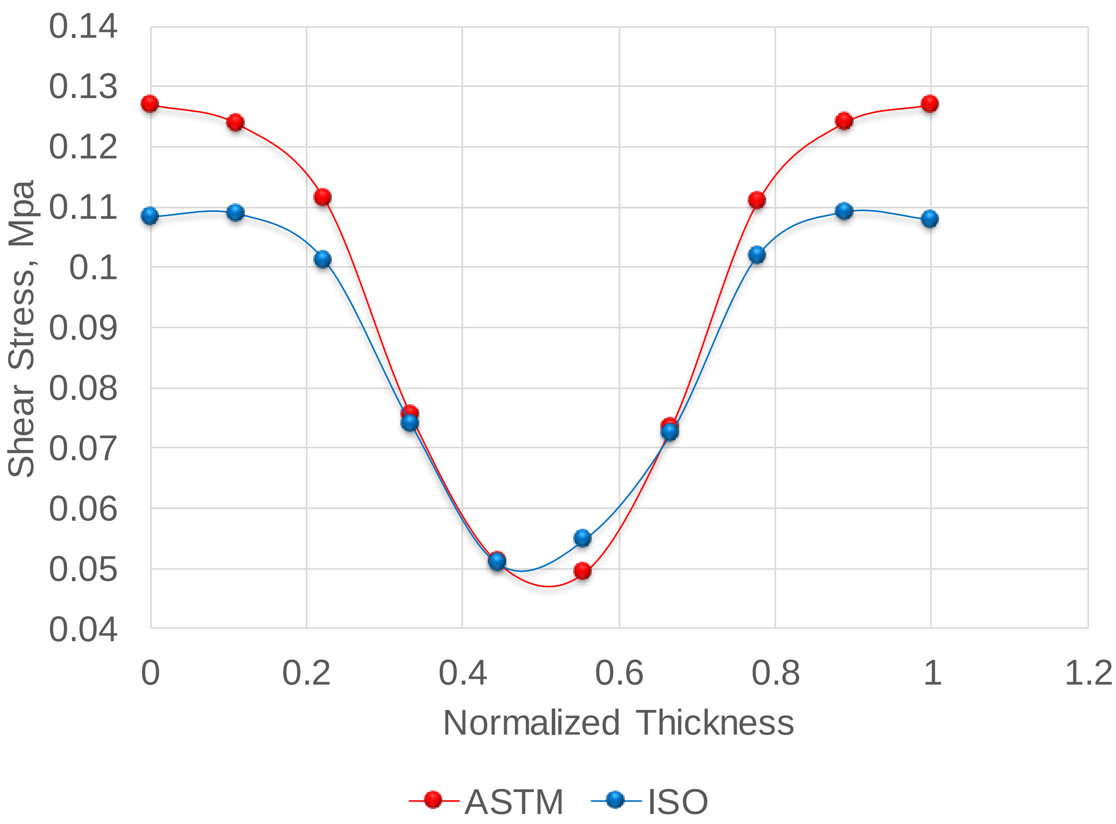


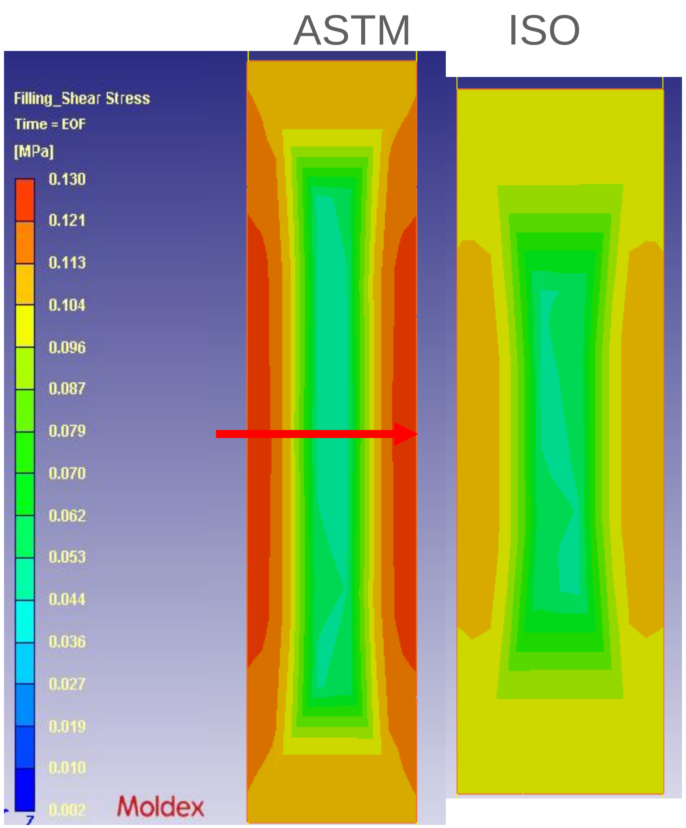


Different temperature distribution in ASTM and ISO bars

- **ISO bar has lower surface temperature**
- **Core of ISO bar takes longer time to cool**

Shear Stress Distribution





- **Shear stress is higher for ASTM**

Predictions Based on First Principles

From the simulation,

- ASTM specimen should show higher flex modulus and yield stress, because of larger thickness of skin layer and shear zone. This is caused by higher shear rate and cooling rate in ASTM specimen.

- ASTM specimen should show higher IZOD especially for ICP. ASTM specimen have more oriented rubber phase and smaller rubber size due to less coalescence.

Design of Experiments

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HPP Resins	MFR
HPP1	3.5
HPP2	1.6
HPP3	8
HPP4	47
HPP5	17
HPP6	3
HPP7	0.3
HPP8	0.1

ICP Resins	MFR
ICP1	6
ICP2	20
ICP3	12
ICP5	75
ICP6	55
ICP7	16
ICP8	20
ICP9	6
ICP10	6
ICP11	5

- **HPP with MFR 0.1 to 50, ICP with MFR 5-75**

Results and Discussion: Flexural Modulus

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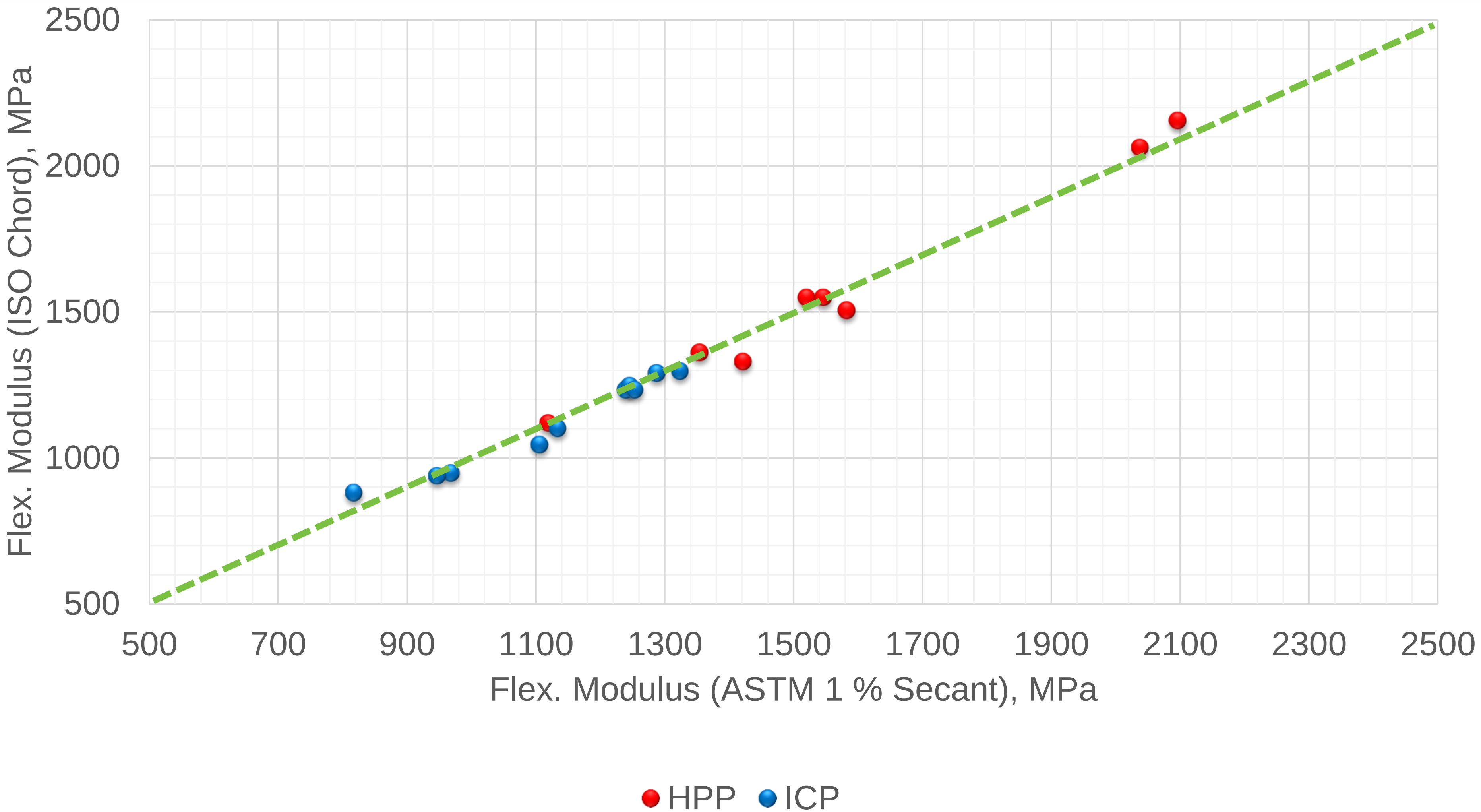

- **Numerical values of flexural modulus equivalent under ISO and ASTM standards**

Results and Discussion: Yield Stress

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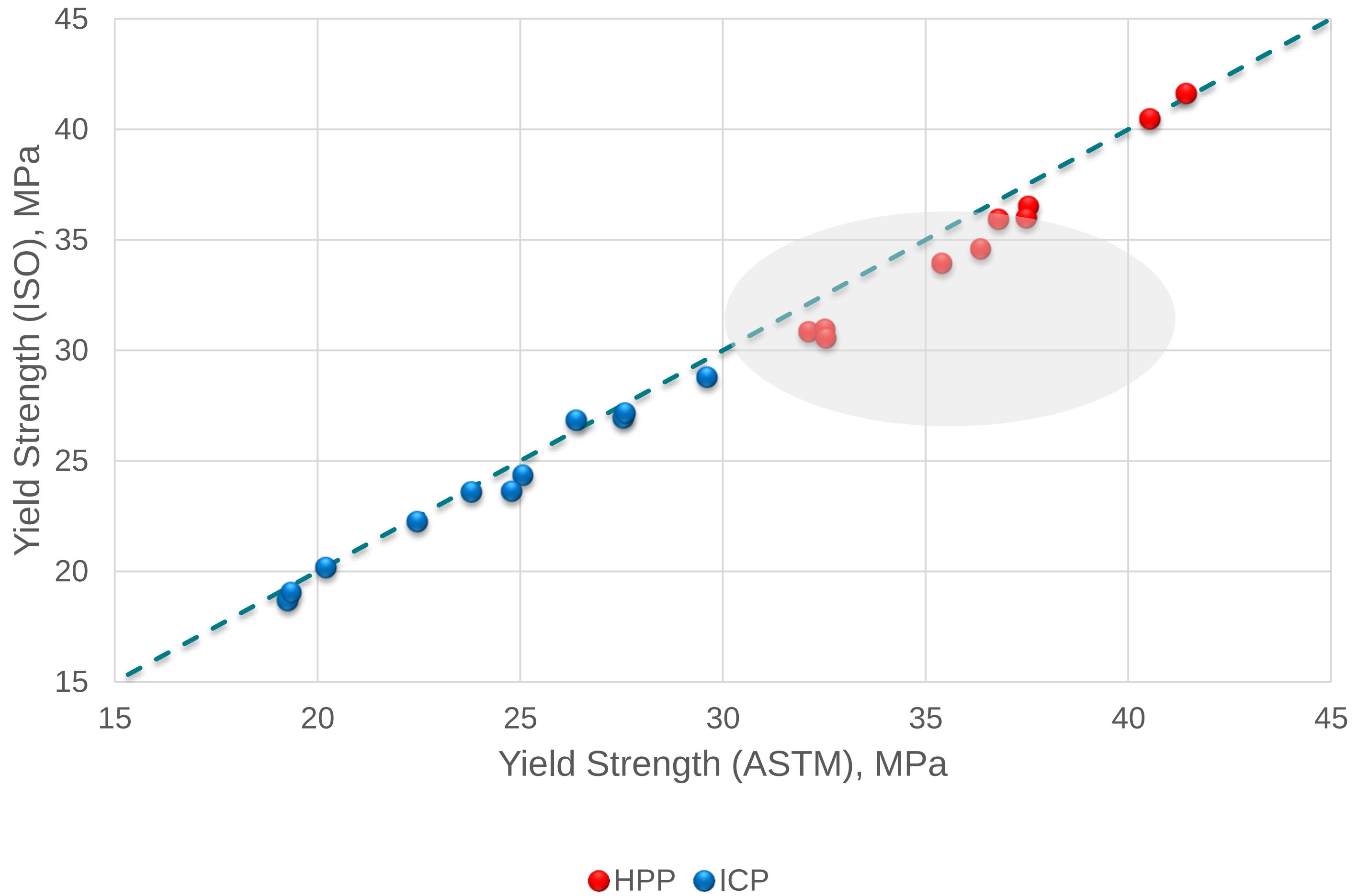
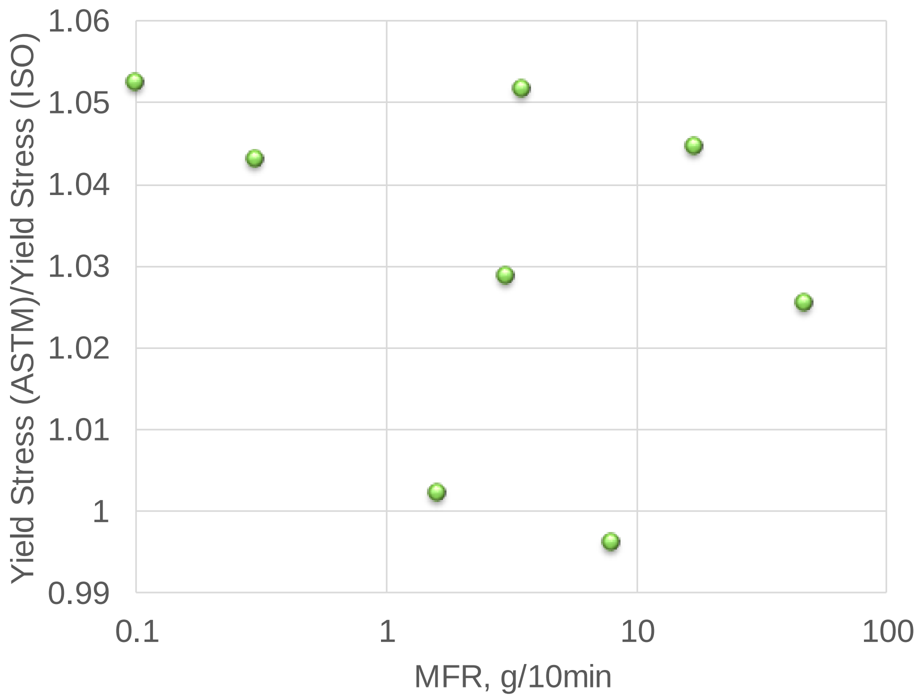
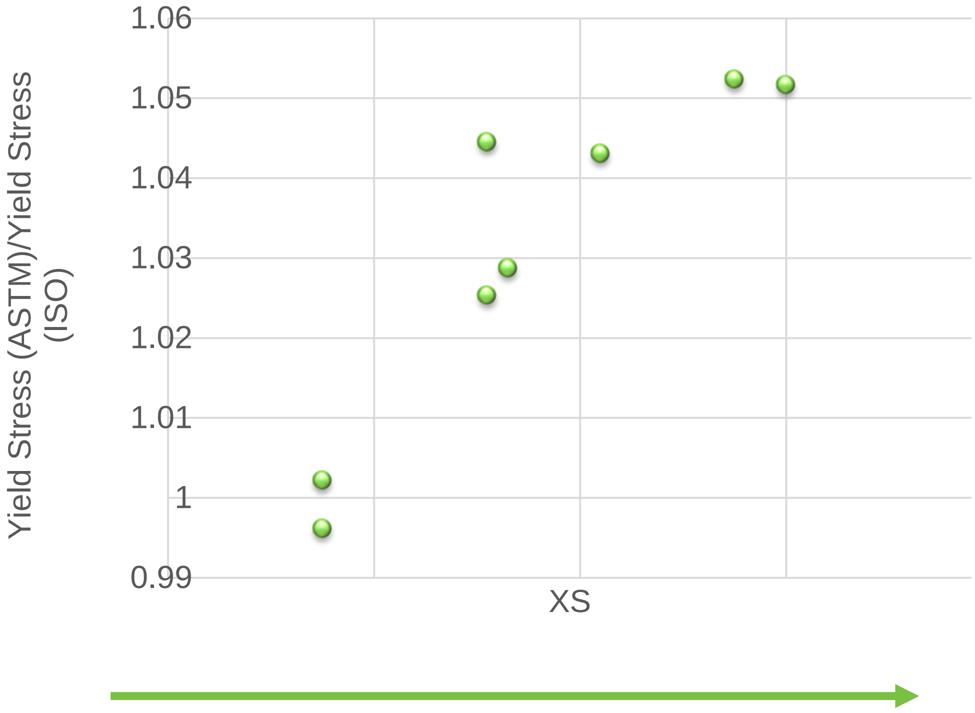


- **Numerical values of yield stress equivalent for ICP**
- **Discrepancy in HPP**

Factors Affecting Yield Stress





- **Variation based on MFR and XS observed.**
- **Multivariate evaluation showed that only XS is relevant variable**
- **When the isotacticity is high, the crystallization rate is faster. Yield strength is less affected by the injection molding conditions.**

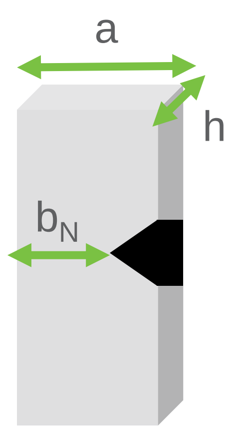
Results and Discussion: Notched IZOD

ASTM D256

- Impact strength is given in J/m
- Impact strength = Ec/h
  - Ec = corrected energy (J) [reading from equipment]
  - h = thickness (m) = 0.0032m
- J/m / 10.2 → kJ/m^2

ISO 180

- Impact strength is given in kJ/m^2
- Impact strength = $Ec/(h*b_N)$
  - Ec = corrected energy (kJ) [reading from equipment]
  - h = thickness (m) =0.004m
  - b_N = length under notch (m)
- kJ/m^2 *8.0 → J/m



Specimen		ASTM (mm)	ISO (mm)
thickness	h	3.2	4.0
Total width	a	12.7	10
Width under notch	b_N	10.2	8

There is no calculation to translate from ASTM to ISO

N. Izod Impact Resistance Comparison

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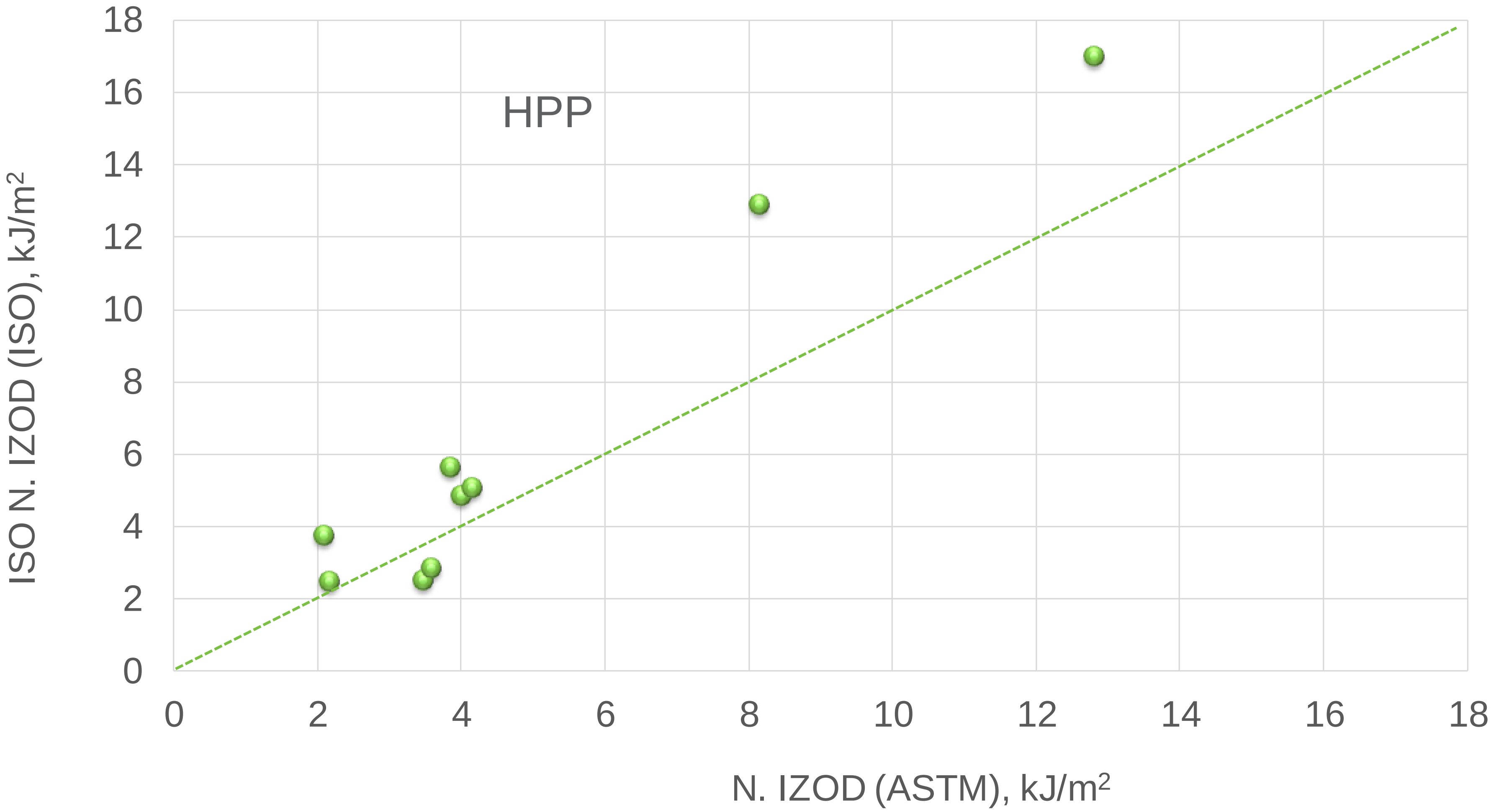

- **ISO bars show different impact resistance compared to ASTM bars**

N. Izod Impact Resistance Comparison

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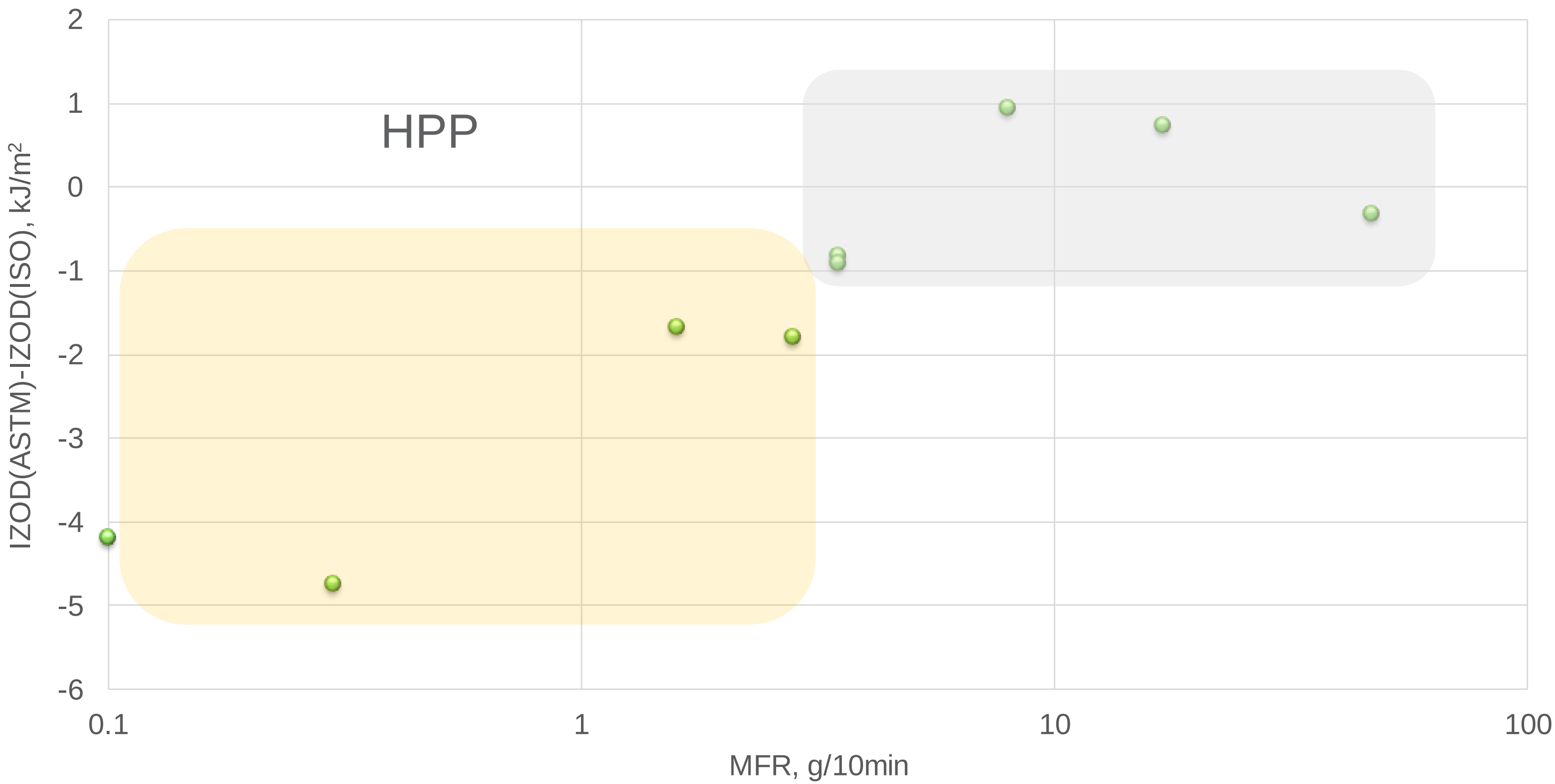

- **ASTM is lower than ISO when MFR is in low range**
- **The difference is possibly due to the orientation and crystal structure in the injection molding**

N. Izod Impact Resistance ICP

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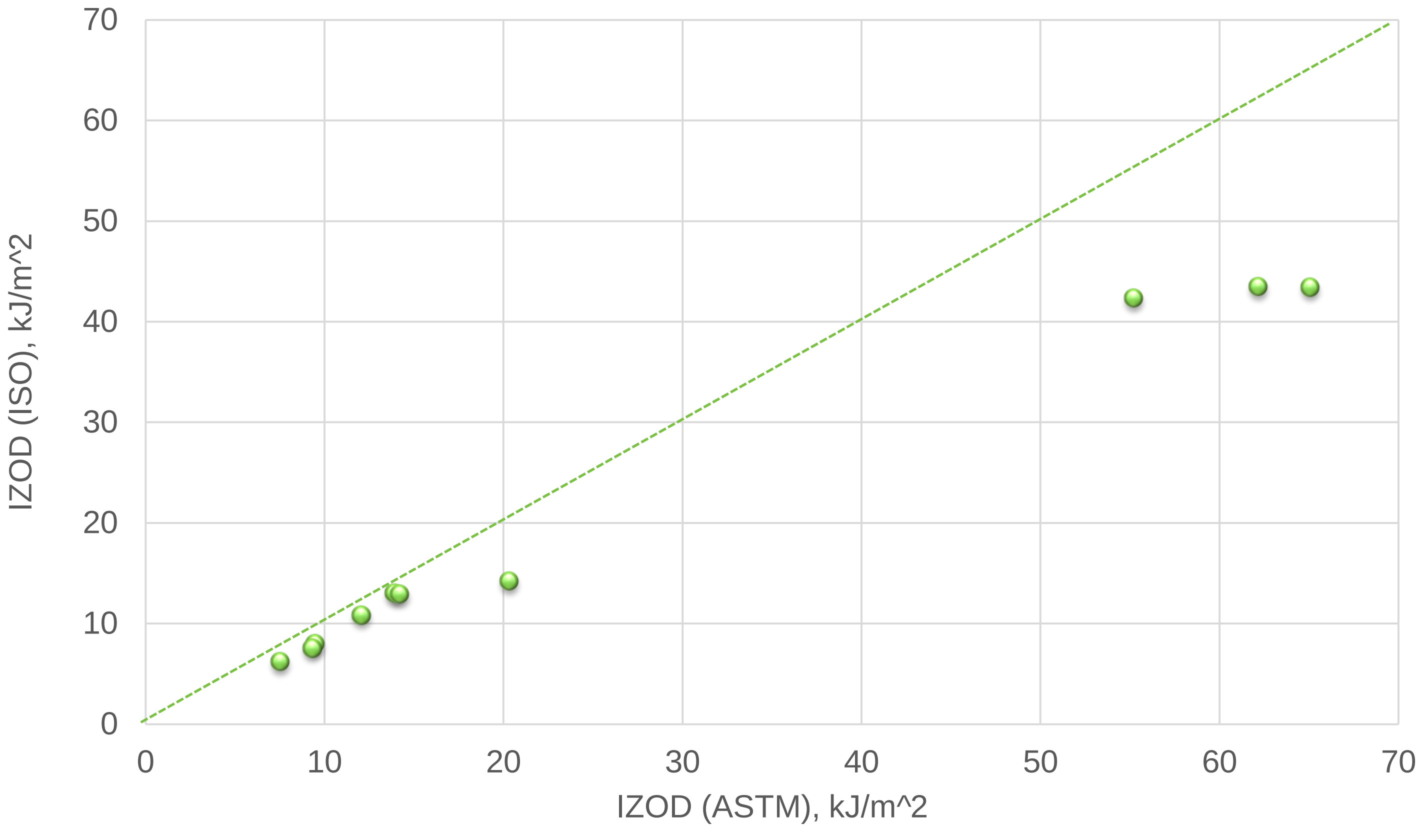

- **Higher ASTM N. Izod impact than ISO**
- **The trend is more obvious with higher impact strength**

Comparison with Technical Data Sheet Data

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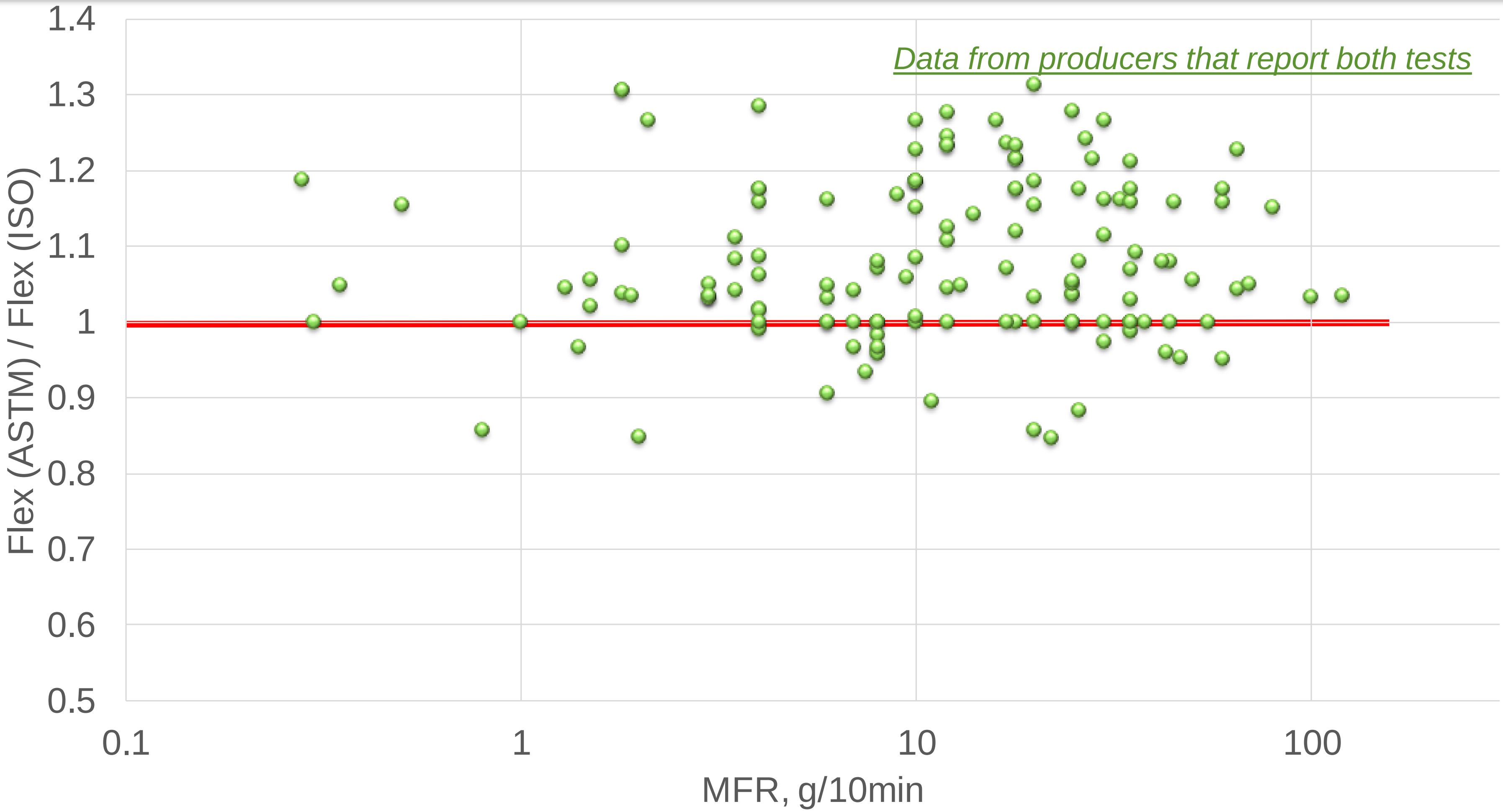

- **Flex(ASTM)/Flex(ISO) between 0.8 and 1.3**
- **>95% of grades are between 0.9 and 1.3**

Comparison with Technical Data Sheets

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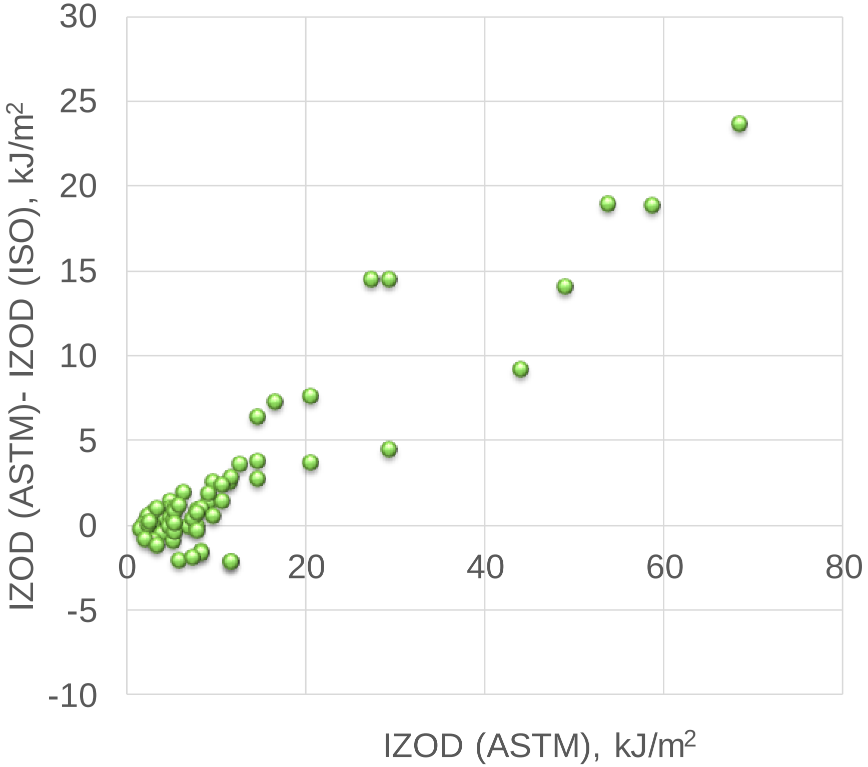

- **Most grade in the range IZOD(ASTM) / IZOD(ISO) 0.7 to 1.7 – Also aligned with findings reported here**
- **Differences increase with IZOD values**

Summary

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	HPP	ICP
Flex. Modulus	ASTM (1% Secant) ≈ISO (Chord)	ASTM (1% Secant) ≈ISO (Chord)
Yield Stress	Medium/High XS, ASTM > ISO; Low XS, ASTM ≈ ISO Overall, ASTM/ISO = 1-1.1	ASTM ≈ ISO
N. IZOD (23°C, kJ/m^2)	Fractional MFR, ASTM < ISO; Medium/High MFR, ASTM ≈ ISO	Low IZOD, ASTM/ISO=1-1.2 High IZOD, ASTM/ISO > 1.2

Critical to understand ASTM and ISO standards to correctly evaluate materials performance… and help our customers develop advanced products…

The End! Thank You.

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