Rizzo Associates Czech, a. s.
ENGINEERS / CONSULTANTS / CM

Utilization of Nanoindentation Methods for Evaluation of the Degradation Rate of Polymeric Materials and Definitions of Condition Monitoring Indicators

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Engineering & Construction Management
Hydro-Nuclear-Fossil
Geotechnical Engineering
Seismic & Structural Engineering
Hydrological & Hydraulic Engineering
Tunnel Engineering
Environmental Engineering & Permitting

Summary

- **Nanoindentation Basics**
- **Condition Monitoring Standards**
- **Several Comments to CM Techniques Defined in Standards**
- **Issues of Relating to CM Indicators**
- **Practice of Nanoindentation Measurements of Polymers**

1. Nanoindentation Basics

Nanoindentation is a technique of the hardness measurement.

Generally, the hardness is a material resistance against intrusion of foreign body.

$$H = \frac{P}{A}$$

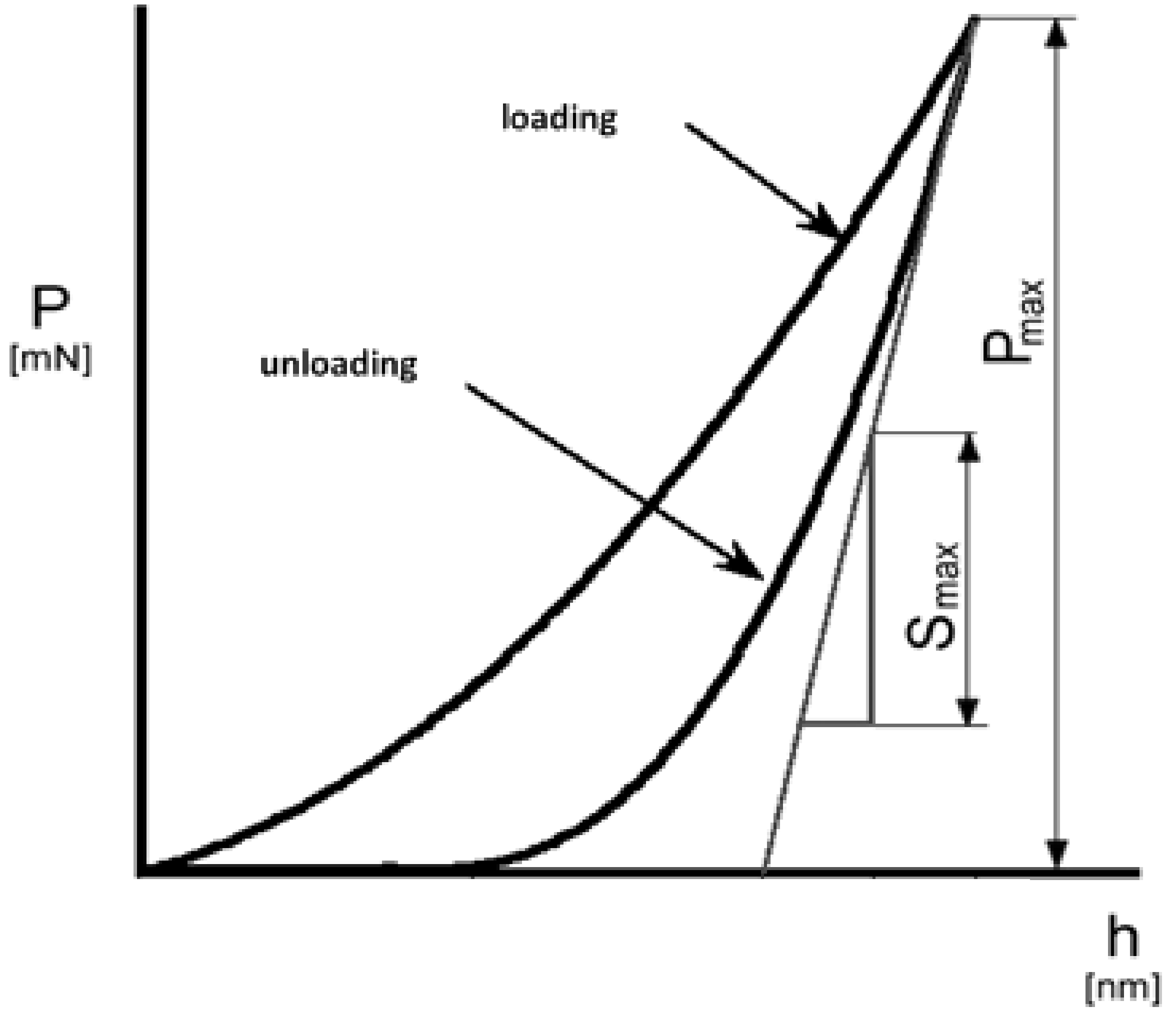
Hardness (H) is expressed by „the force (P) – indentation area (A)“.

Hardness expressed by „the force-strain curve (indentation curve)“ parameters is **Dynamic Hardness**.

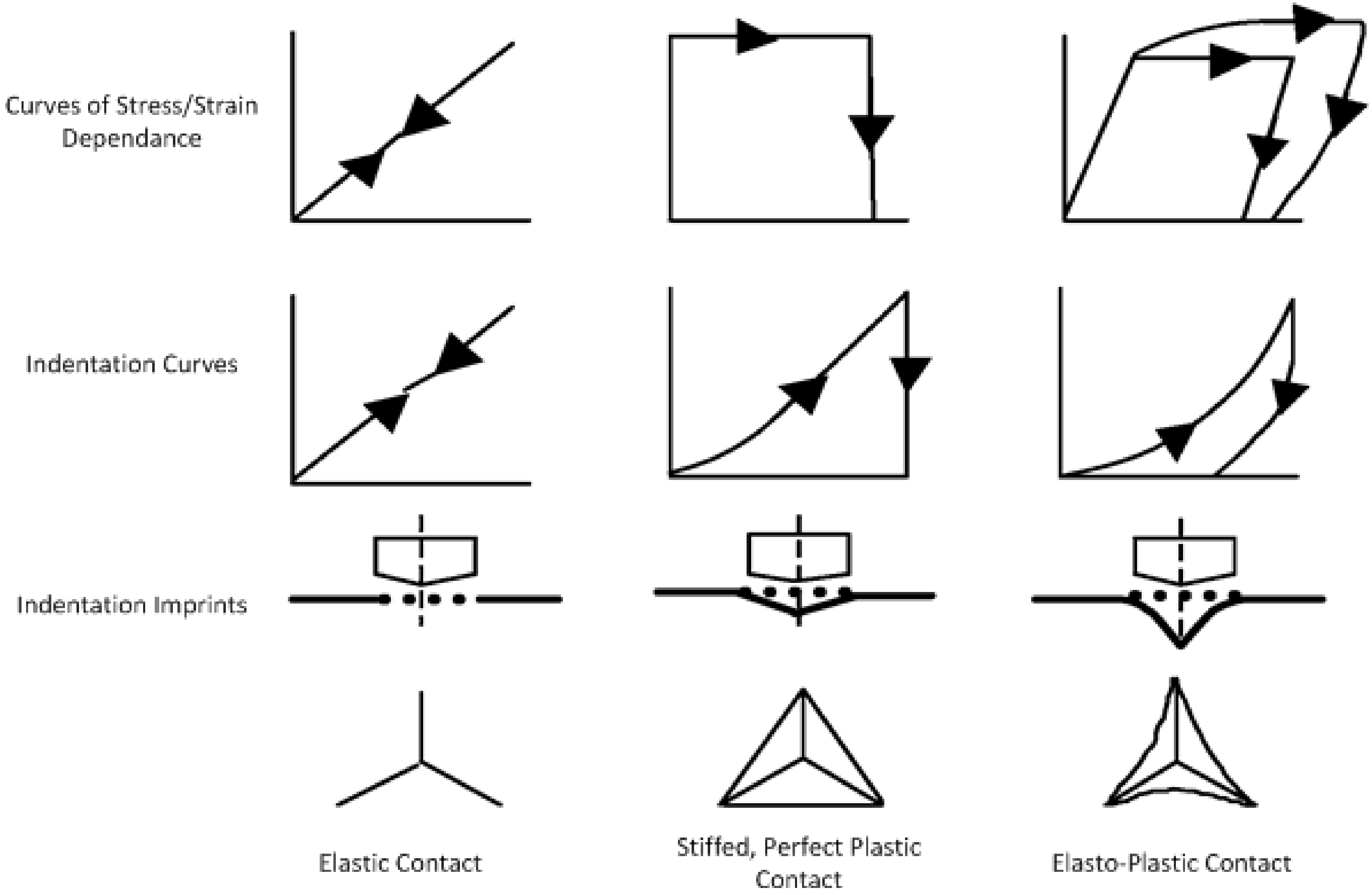
In general, indentation measurement is using for definition of hardness. There is distinguished the type of hardness in accordance of applied load force:

- Macro-hardness (< 50 N),
- Micro-hardness (< 30 N),
- Nano-hardness (< 300 mN)

1. Nanoindentation Basics



1. Nanoindentation Basics



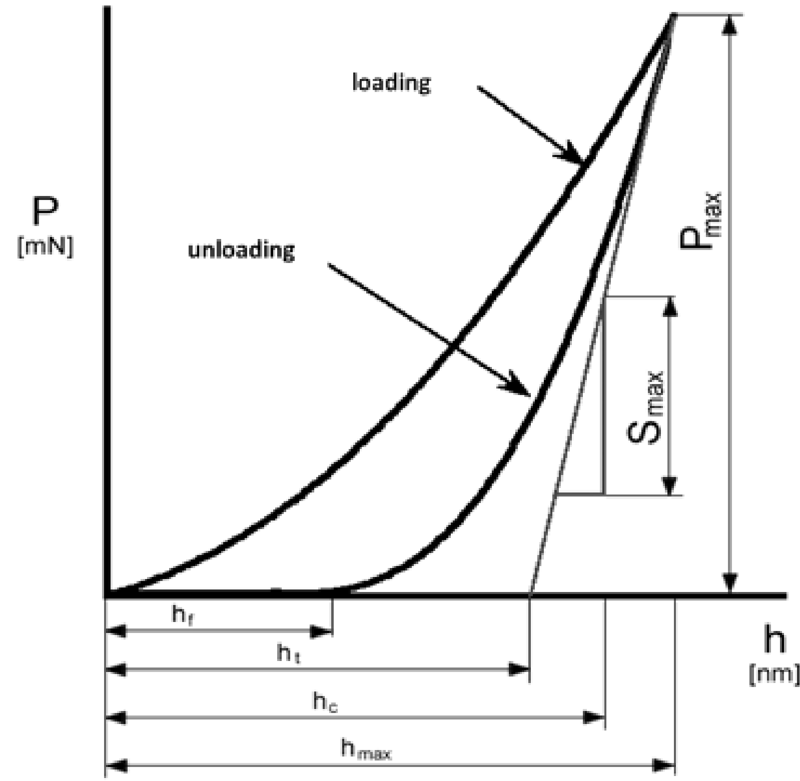
1. Nanoindentation Basics

Dependence between penetration depth *h* and loading force *P* can be expressed by Oliver-Phar relation:

$$P = \alpha \cdot \left(h - h_f\right)^m$$

Contact conditions can be expressed by Sneddon-King relation, that defined the Young‘s modulus:

$$S = \frac{dP}{dh} = \beta \cdot \frac{2}{\sqrt{\pi}} \cdot E_r \cdot \sqrt{A}$$

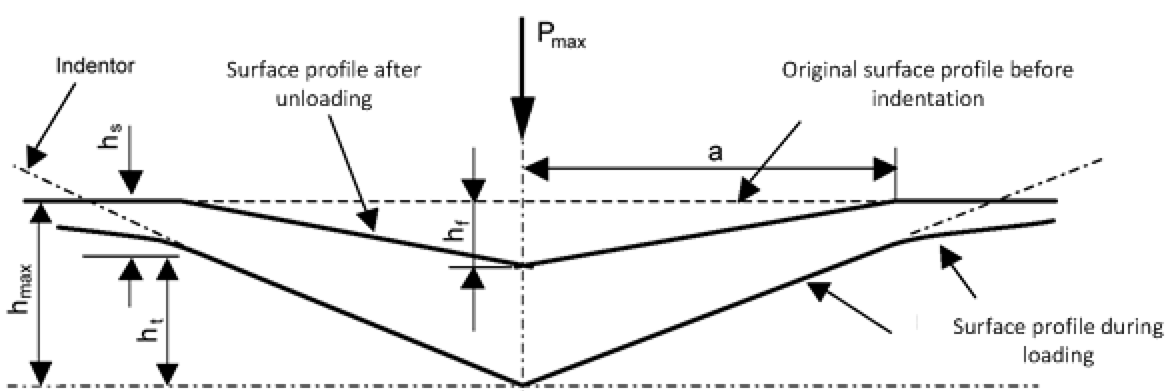


h_{max} - Maximum identor penetration depth

h_f - Penetration depth of the indentor after unloading

h_t - Depth correspondents with intersection of the stiffness tangent at maximum loads and depth

h_c - Calculated contact depth



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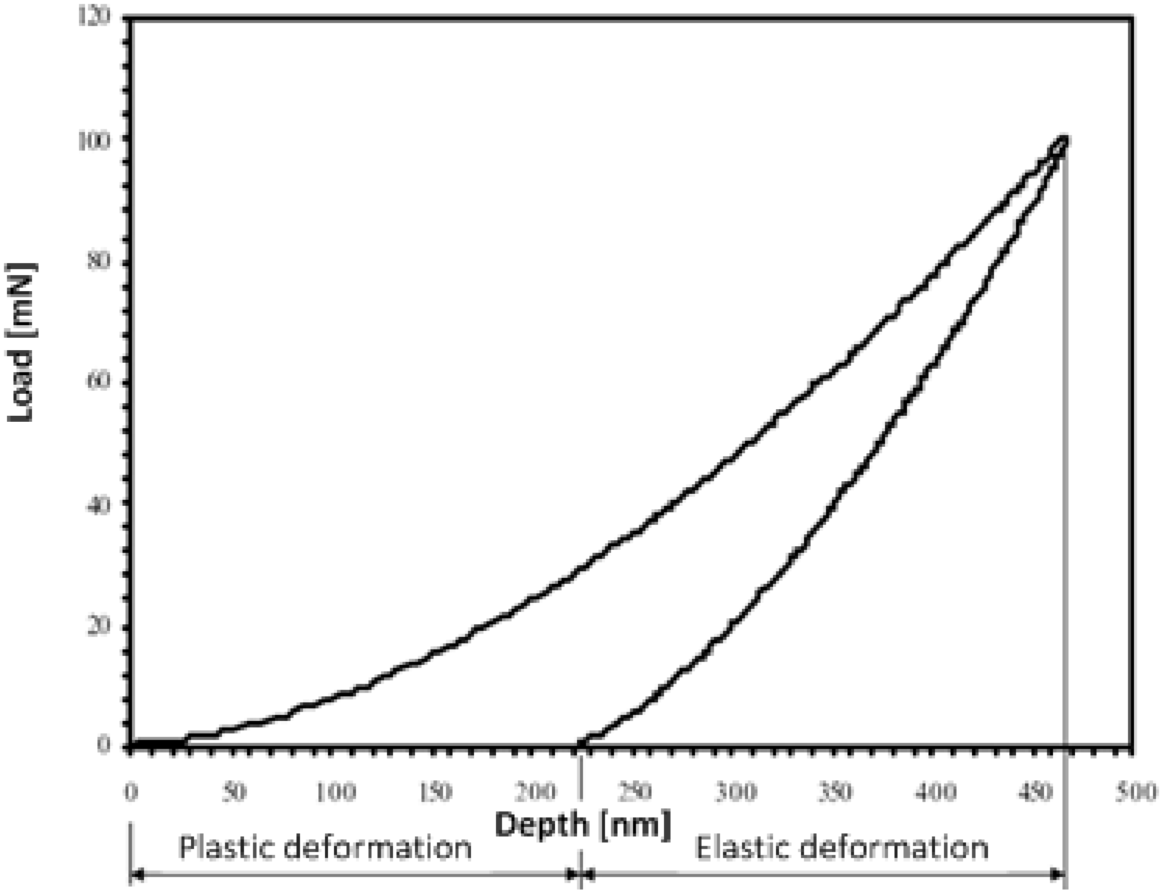
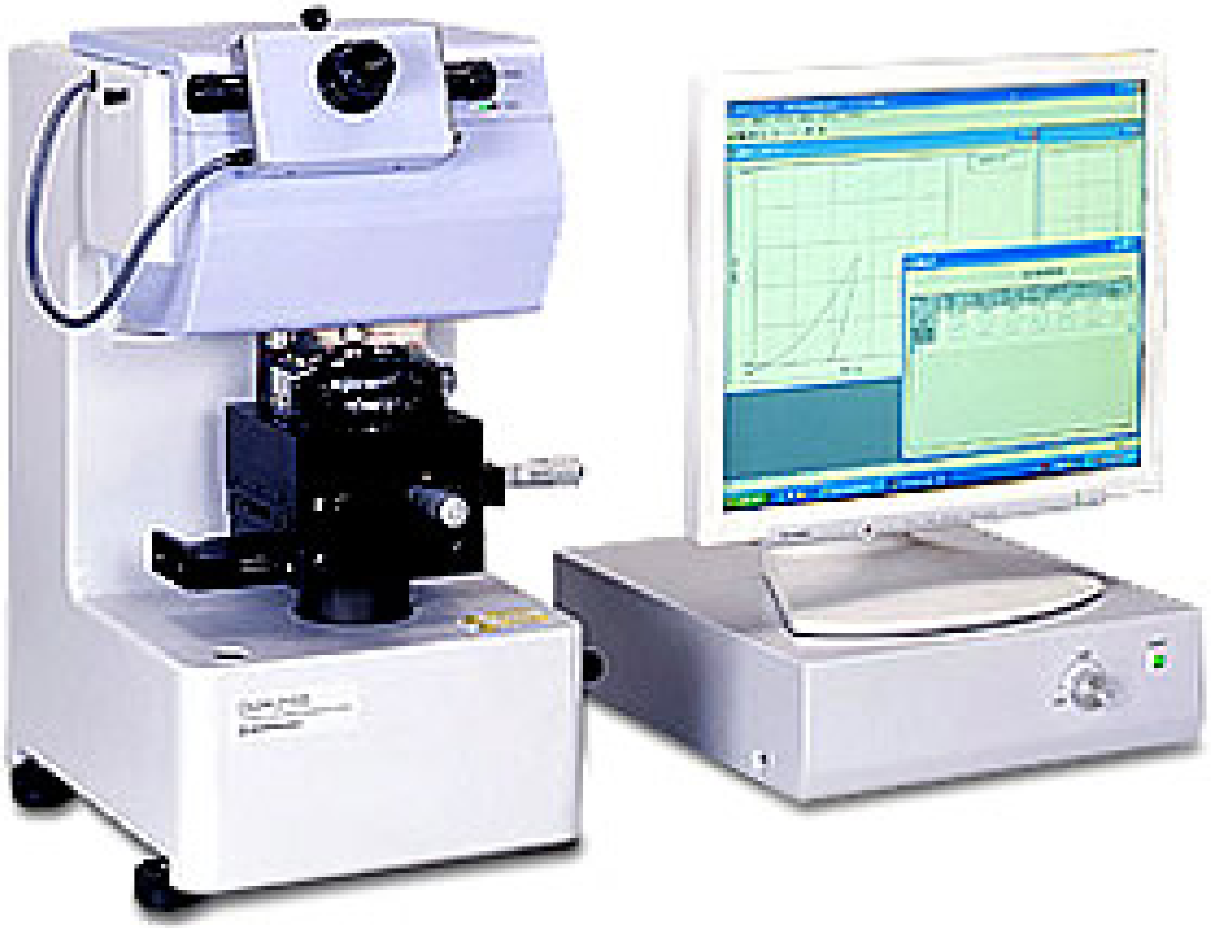


Alternations of ratio of plastic – elastic deformation can be also simply recognized:

$$R = \frac{d_e}{d_p}$$

PCR

1. Nanoindentation Basics



2. Condition Monitoring in Standards

IEC/IEEE 62582

IEEE Std 323-2003

2.1 Condition Monitoring in Standards - Comments to CM Indicators

IEEE Std 323-2003,
Sect. 5.3 Condition monitoring

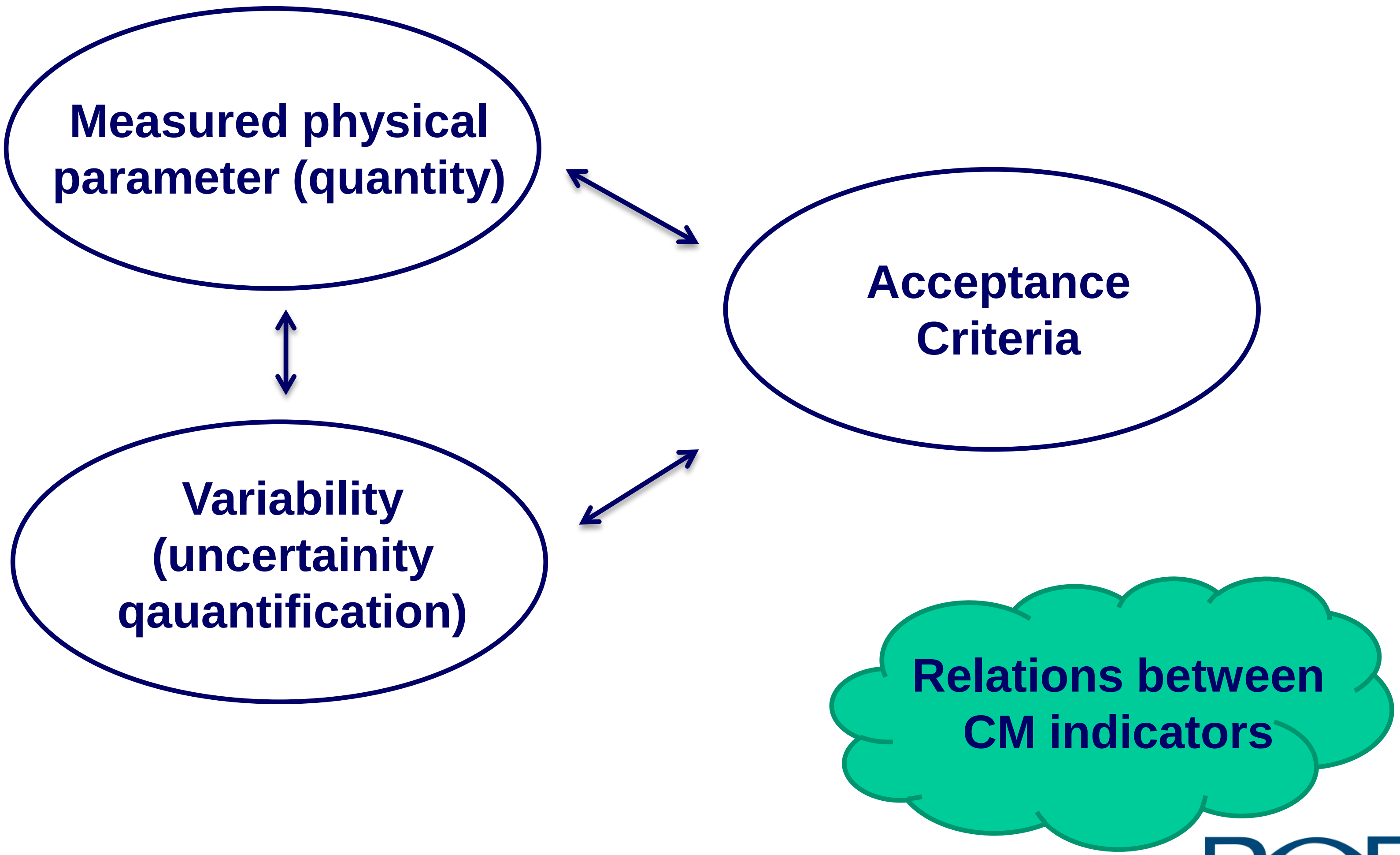
Condition monitoring may be used in place of a qualified life to determine if qualified equipment is suitable for further service. Condition monitoring for equipment qualification purposes <u>monitors one or more condition indicators</u> to determine whether equipment remains in a qualified condition. <u>The trend of the condition indicator</u> is determined during the performance of age conditioning of the test specimen during qualification testing. <u>The condition indicator must be measurable, linked to the functional degradation of the qualified equipment, and have a consistent trend from unaged through the limit of the qualified pre-accident condition</u>. Condition monitoring may be used with or independently from the concept of qualified life. As the qualified equipment approaches the end of its theoretical qualified life, periodic condition monitoring may be implemented to determine if actual aging is occurring at a slower rate, and if further qualified service is possible based on the condition monitoring results.

2.1 Condition Monitoring in Standards - Comments to CM Indicators

IEC/IEEE 62582-1:2011
Sect. 4 Condition indicators,
4.1 General

Condition monitoring should only be applied if there is a known relationship between the ageing degradation of the component monitored and the degradation of the equipment's safety function. This relationship should be established during equipment qualification or a separate duplication of the aging with knowledge of the relationship during design bases events. The relationship should take into account any diffusion limited rate effects that occur during accelerated ageing with high acceleration factors.

3. CM Indicators Hassel



4 Condition Monitoring Methods – Indentor Modulus and Elongation at Break

Indentor Modulus

- High variability between different cable construction types
- Acceptance criteria is missing
- Using just for the cables (limitations for cables of certain dimensions)

Elongation at Break

- EtB is widely accepted CM indicator
- Acceptance criteria well recognized, e.g.
  - EtB <50% end point criterion for safety cables
  - EtB <25% end point criterion for other cables

  NOTE: See International Nuclear Plant Electrical Cable Ageing Management Symposium, France, September 17-19, 2013
- Questionable reproducibility because several techniques are introduced in the stanard

4.1 Condition Monitoring Methods – Indentor Modulus, Comments to CM Indicators

IEC/IEEE 62582-2:2011
Sect. 6.2 Reproducibility

Indenter modulus values can be influenced by variability in <u>specimen dimensions</u>, temperature and humidity of the specimen, stabilization of the specimen, and contamination of the specimen. <u>The influence by variability in the specimen dimensions is typically the case for measurements on cables</u>, where the measurement point may be situated above a cavity beneath the jacket surface. <u>The cross - section of typical cable core insulation may differ substantially from that of an ideal tube and can result in variability in the measured values of indenter modulus</u> depending on where the measurement is made. These variations tend to be localized. Measurements shall be taken at several points on the equipment to compensate for these local variations.

4.1 Condition Monitoring Methods – Indentor Modulus, Comments to CM Indicators

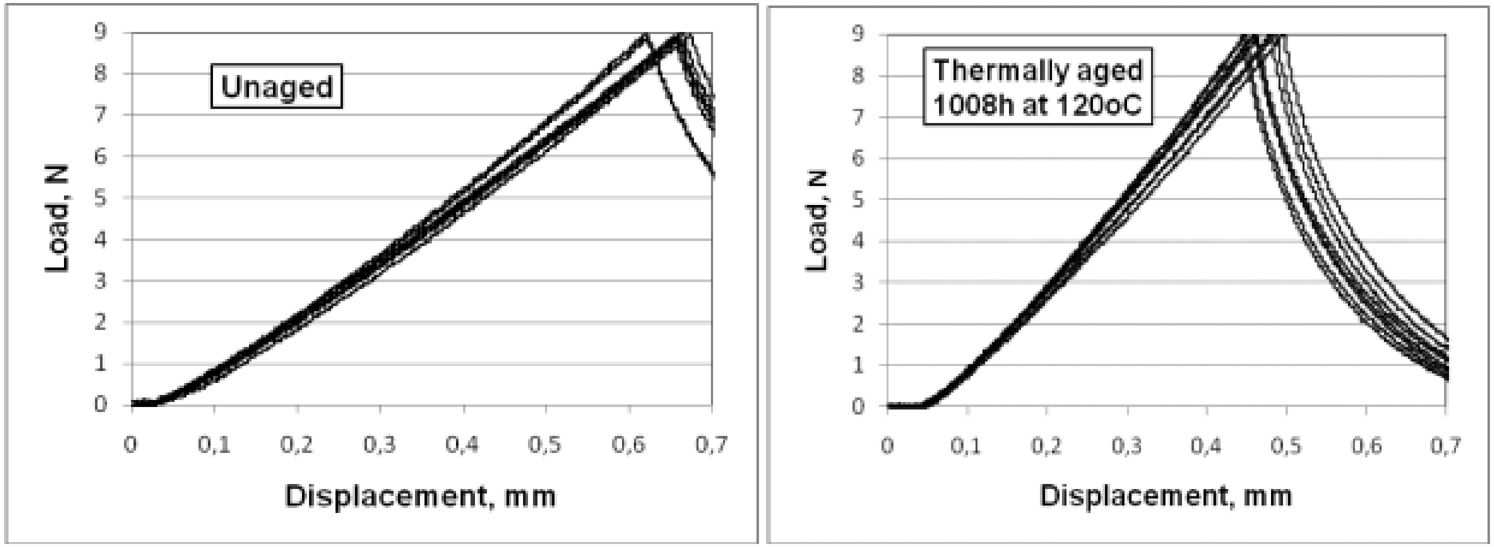
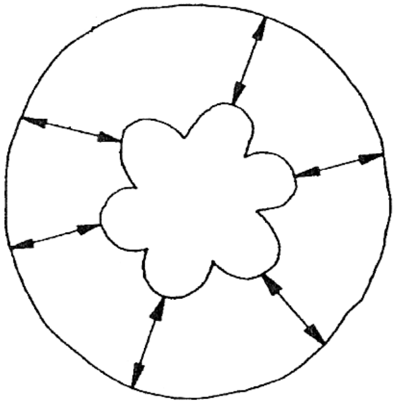


Figure A.1 — Example of local variation of indenter modulus due to variation in equipment dimensions



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4.1 Condition Monitoring Methods – Indentor Modulus, Comments to CM Indicators

Equipment ID	274 Cable FSSR 7X1
Material	Jacket: CSPE (2 mm) blue; Insulation and conductors: 7 core EPDM on stranded Cu grey (0,7 mm)

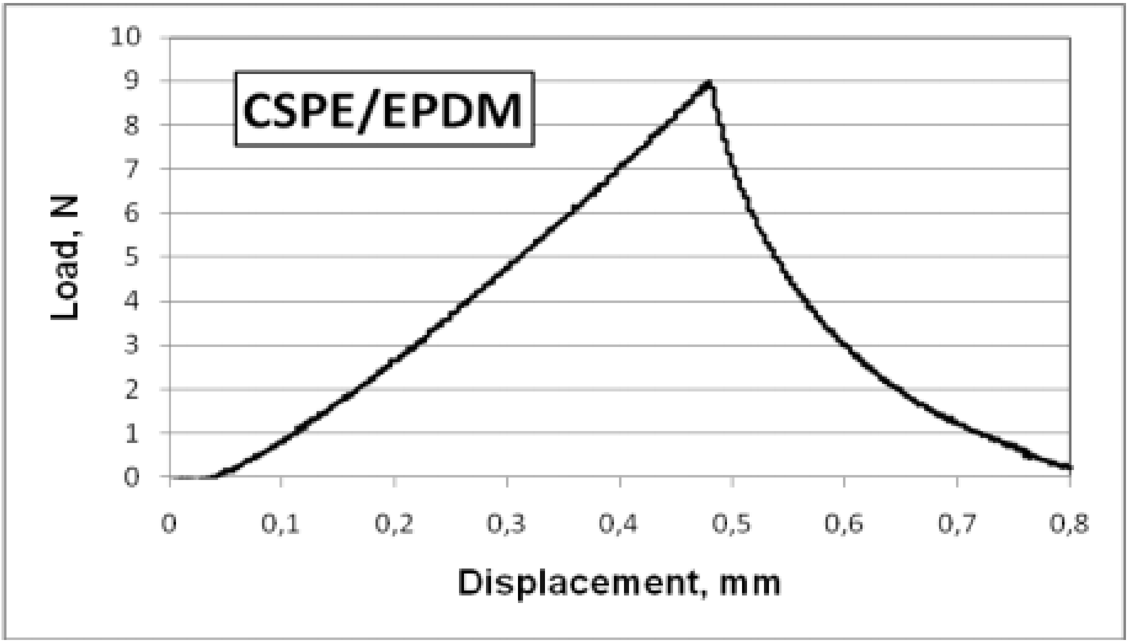
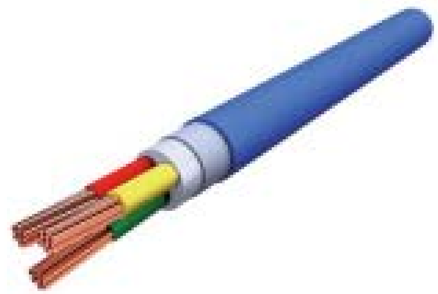


Figure B.1 — Example of measured force vs displacement

4.1 Condition Monitoring Methods
– Indentor Modulus, Comments to CM Indicators

MV Power cables



LV Power cables

Control cables

Instrumentation cables

Communication cables

4.2 Condition Monitoring Methods – Elongation at break, Comments to CM Indicators

IEC/IEEE 62582-3:2012
Sect. 4 General description

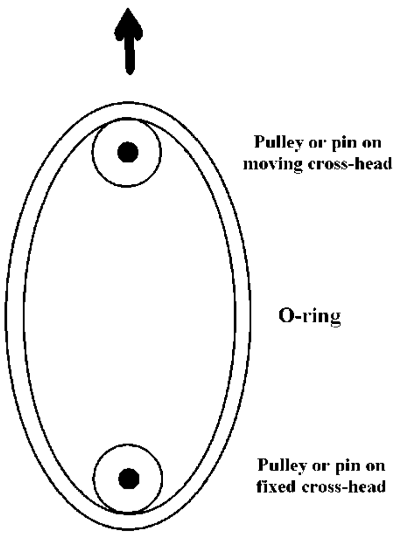
NOTE - Elongation at break rather than tensile strength is used because for some polymers, particularly thermoplastics, the strength may remain consistently equal to the yield strength after ageing even when the elongation has decreased to < 50% absolute.

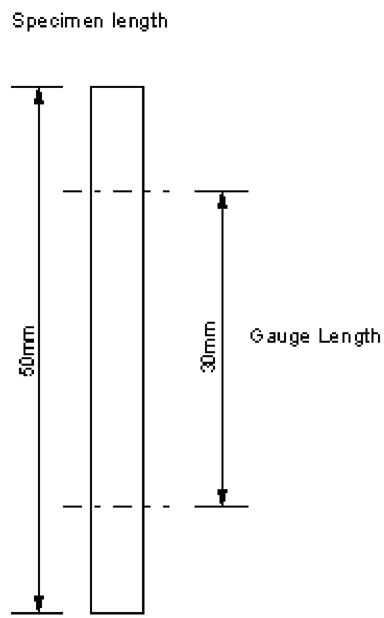
Sect. 5 Applicability and reproducibility
The method is related to the long chain molecular structure of the polymer. As degradation proceeds, changes in the molecular structure occur as a result of cross-linking, chain scission, oxidation and other degradation mechanisms. These changes usually decrease the elongation at break.

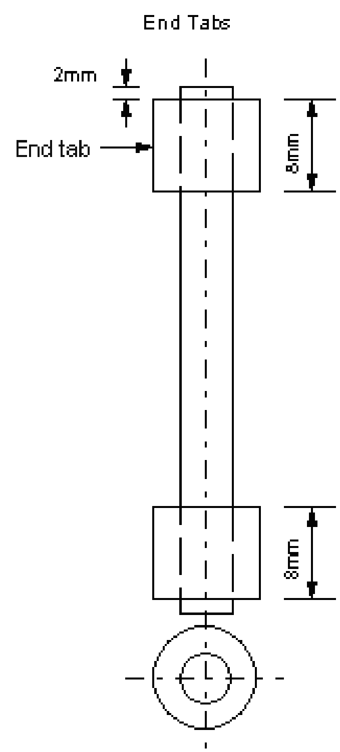
This method is primarily suited to samples taken from equipment that are based on thermoplastic or elastomeric polymers. The method is generally not suitable for fibre reinforced polymers or resins such as epoxides.

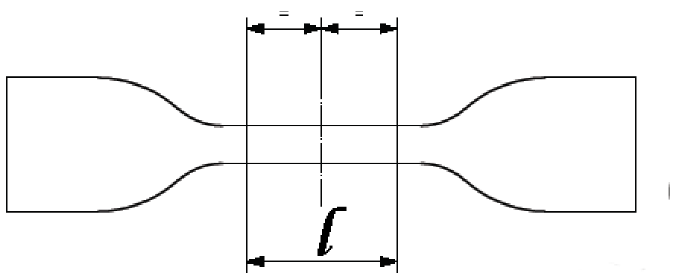
4.2 Condition Monitoring Methods – Elongation at break, Comments to CM Indicators

A test specimen is extended along its longitudinal axis at constant speed until the specimen fractures. During the test, the load sustained on the specimen and its elongation are measured. For this standard, elongation at break is the measured parameter.







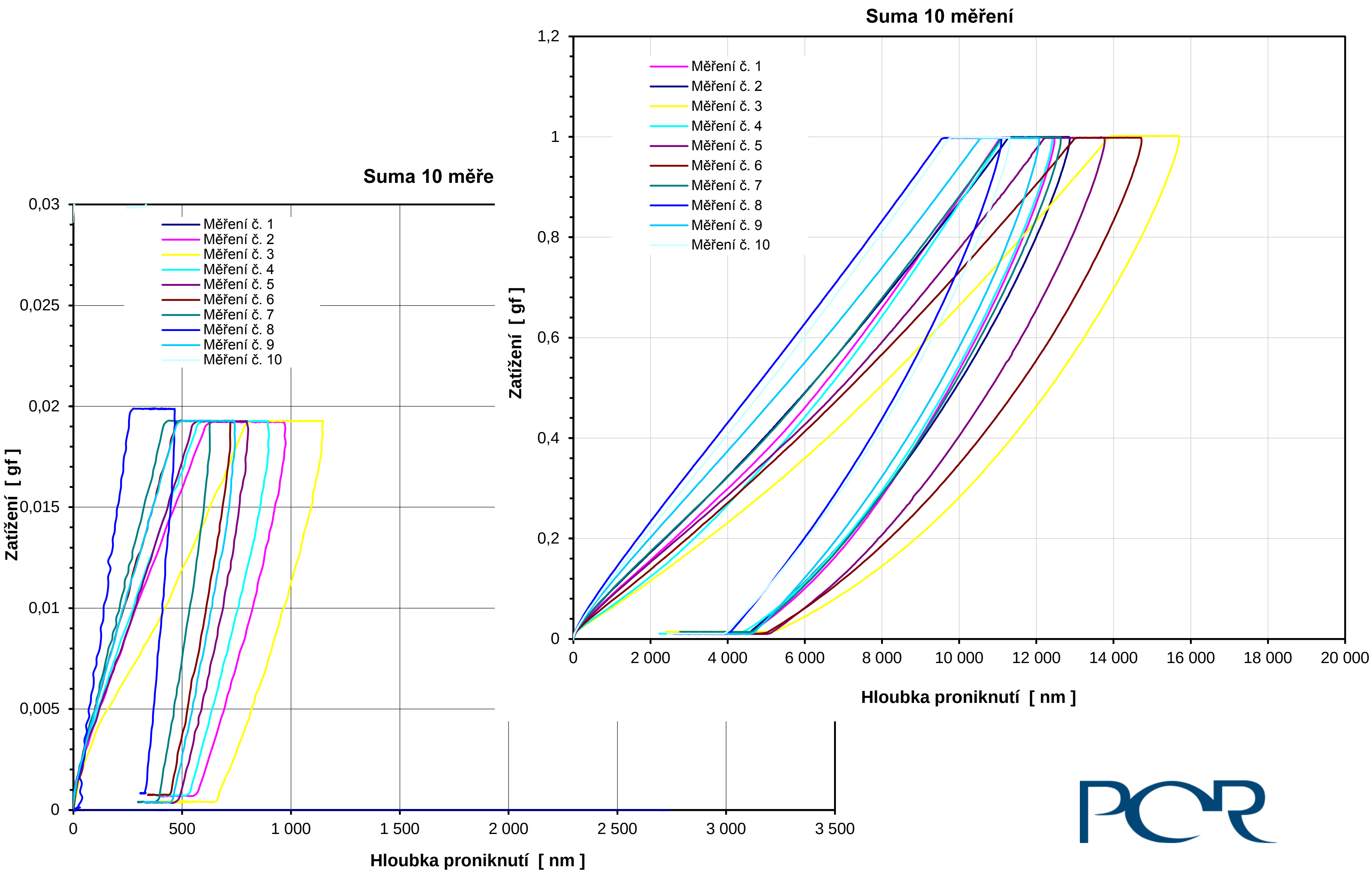


Our Mission in Nanoindentation Polymers Testing

The collaboration of Rizzo Czech Associates and West Bohemian University (driven by our passion).

- **Lowering of variability implicit in using of measurement technique of CM indicator (mechanical quantities)**
- **Convergence of Indenter Modulus and EtB measurement technique defined in IEC/IEEE 62582 standards**
- **Definition of the common-basis in an intention of „mechanical quantities“ of CM Indicators and they correlation with „engineering properties“**

5. What We Are Doing

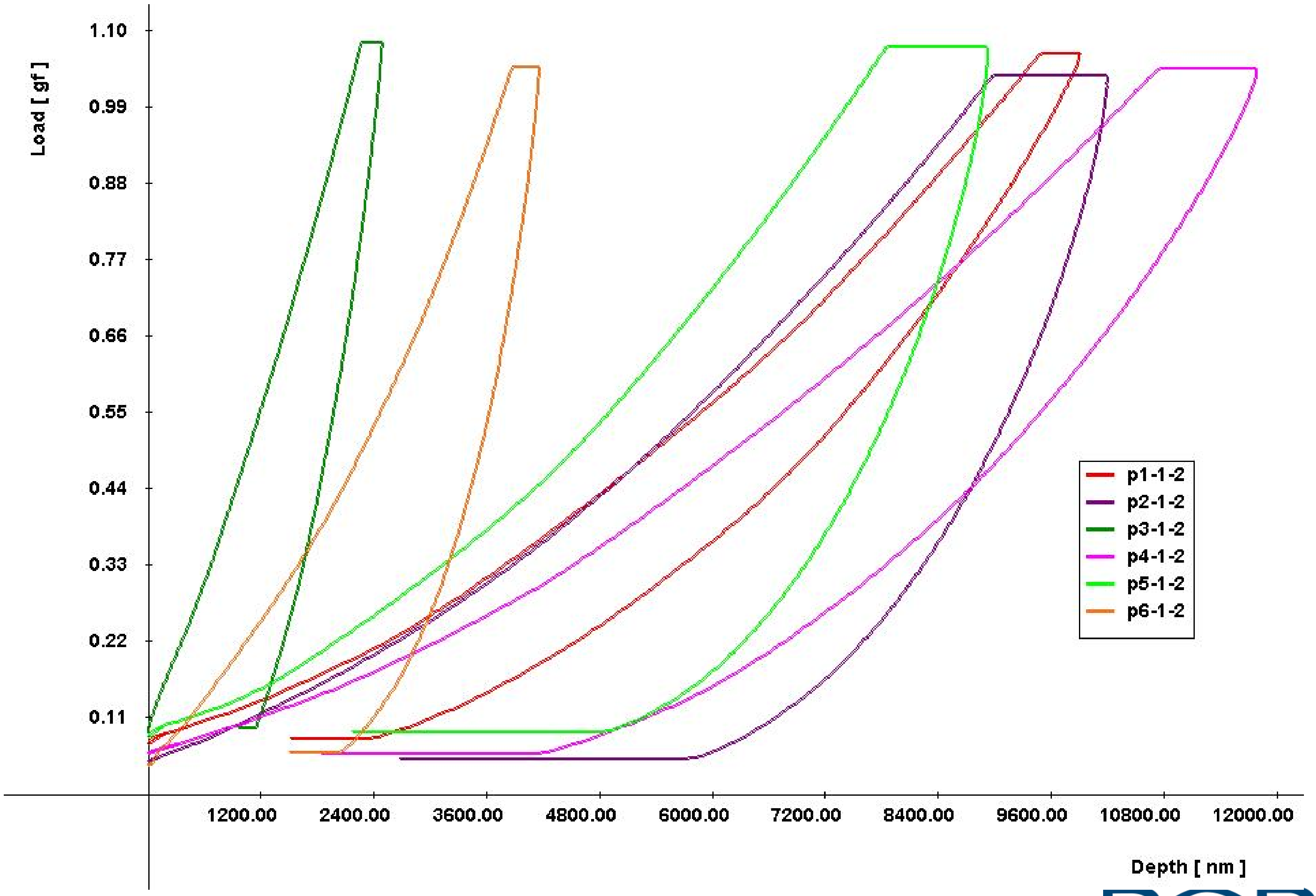


5. What We Are Doing

Označení typu vzorku	Název	Výrobce	Báze	Použití
A	Ongrolit LEV 720	BorsodChem Rt.	PVC	izolace vodičů a kabelové pláště
B	Catapyrric SX407K:CM422	AEI Compounds Limited	silan - sesíťovatelný polyethylen	izolace vodičů a kabelové pláště
C	Megolon S504	Scapa Polymerics Ltd	nespecifikovaný termoplast	kabelové pláště
D	AEI SX408:CM401	AEI Compounds Limited	silan - sesíťovatelný polyethylen	izolace vodičů a kabelové pláště
E	Dytron XL 7300-82	Advanced Elastomer Systems	ethylen-vinyl acetát	izolace optických, řídících, telefonních kabelů, ohebná jádra kabelů

Pilot study accelerated thermal ageing conditions (applied for all samples): 800 hod. / 100°C

5. What We Are Doing



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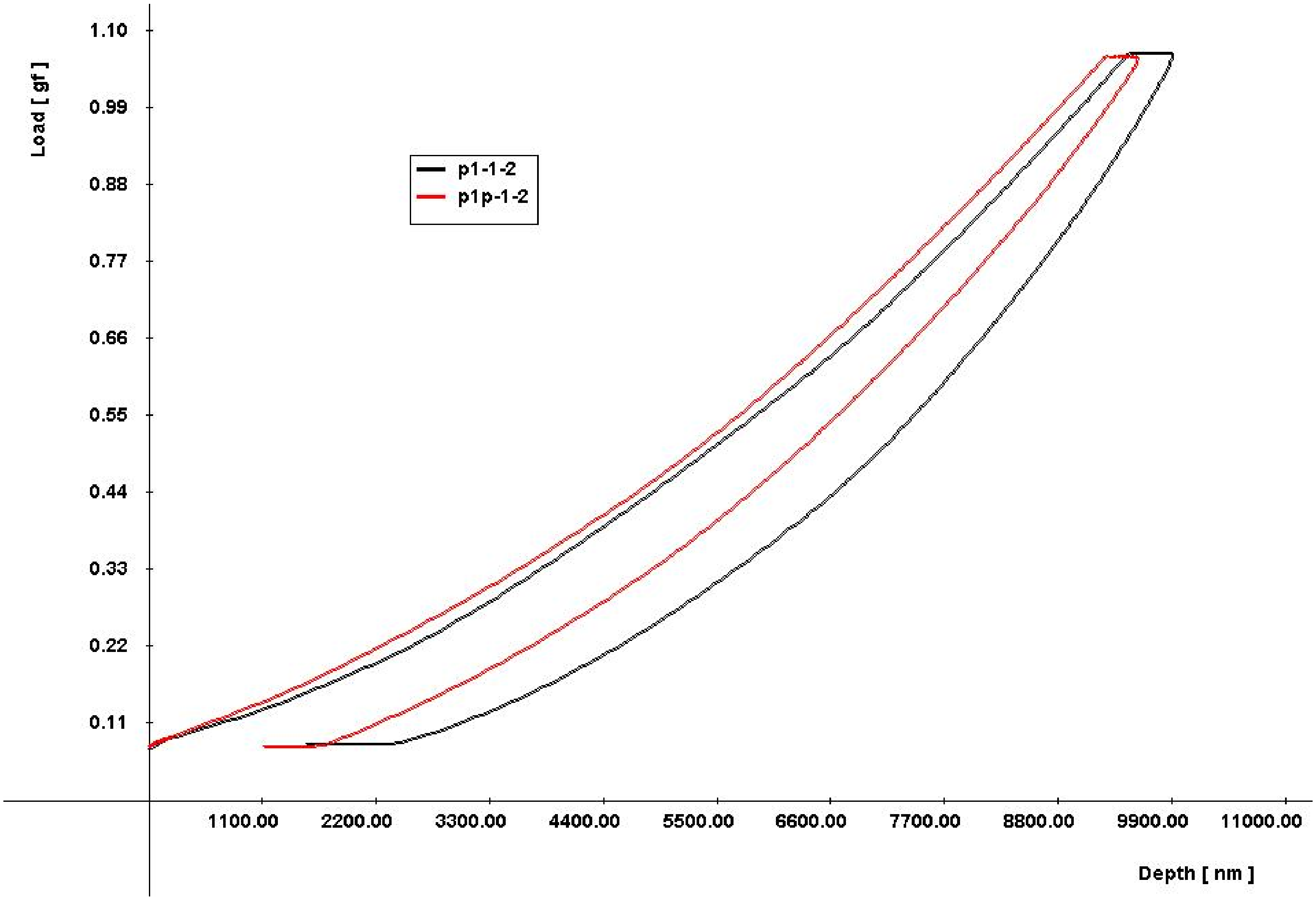

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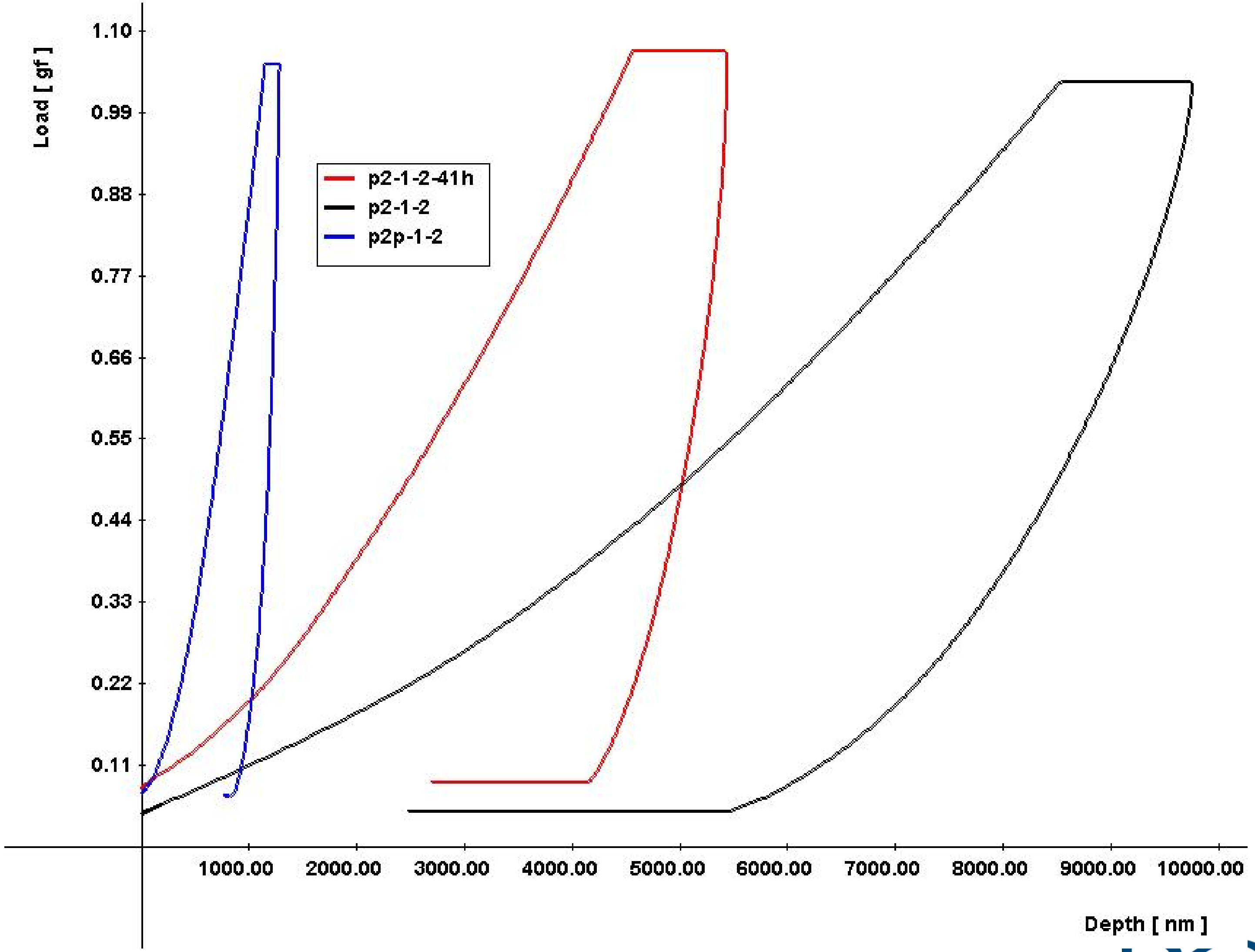

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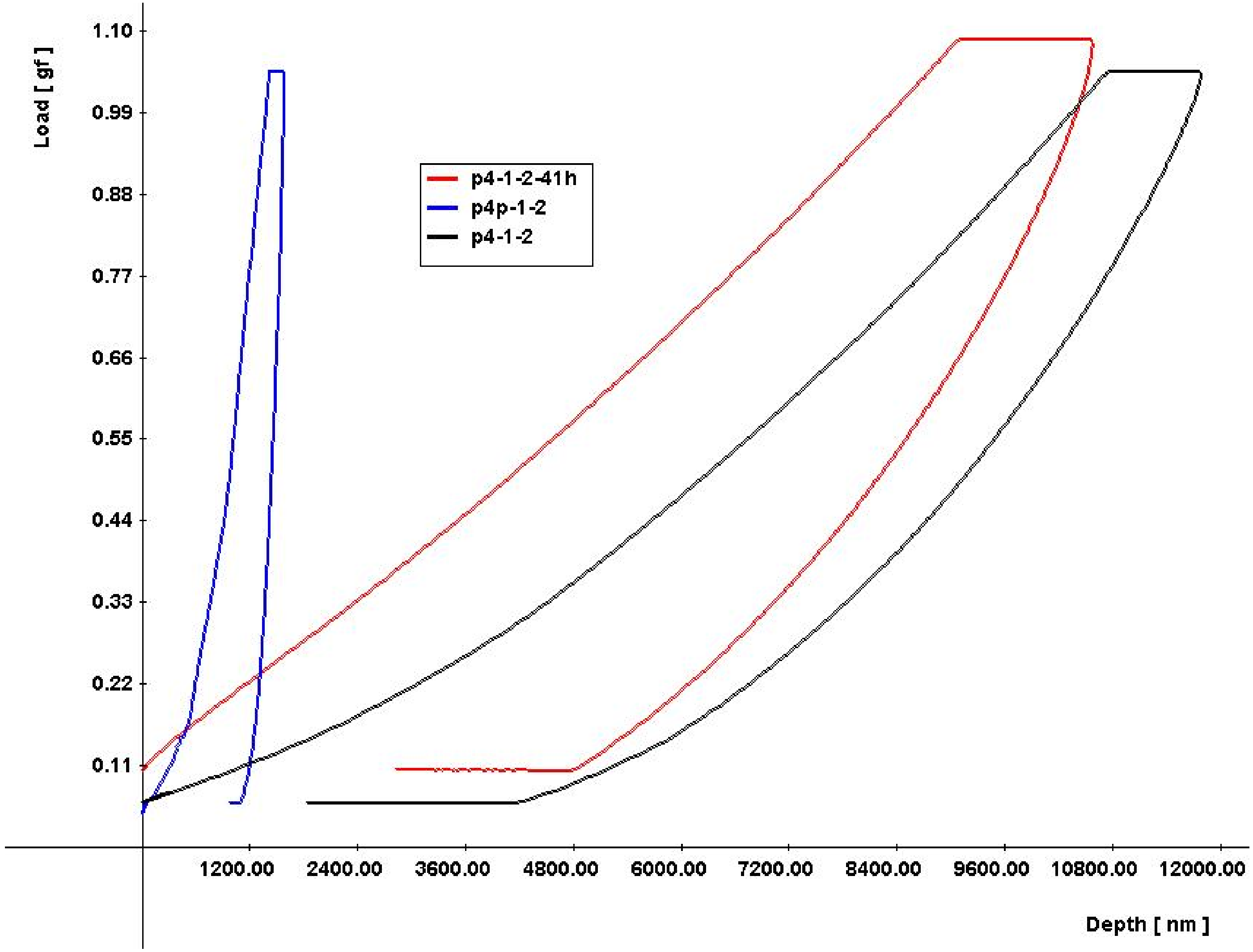

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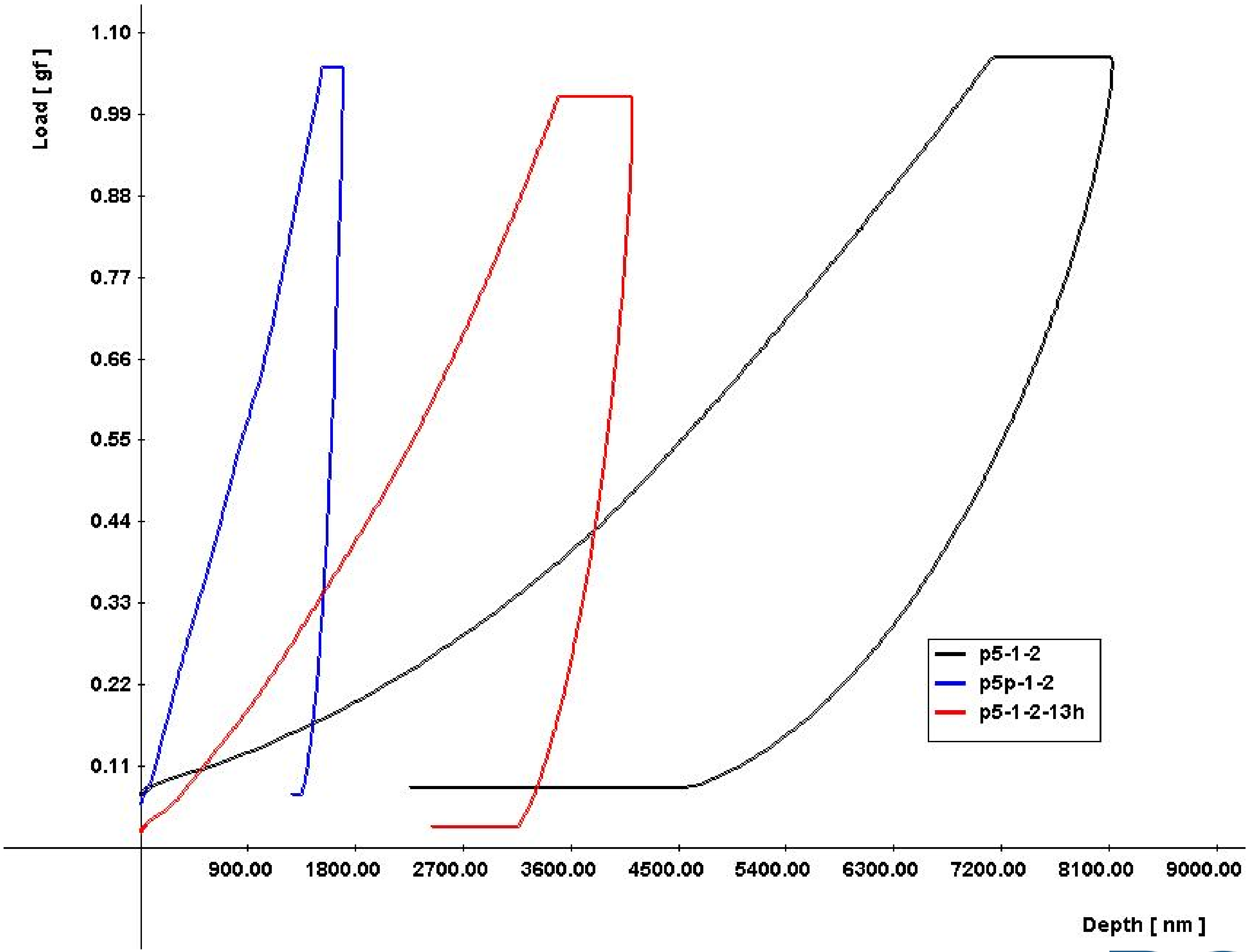

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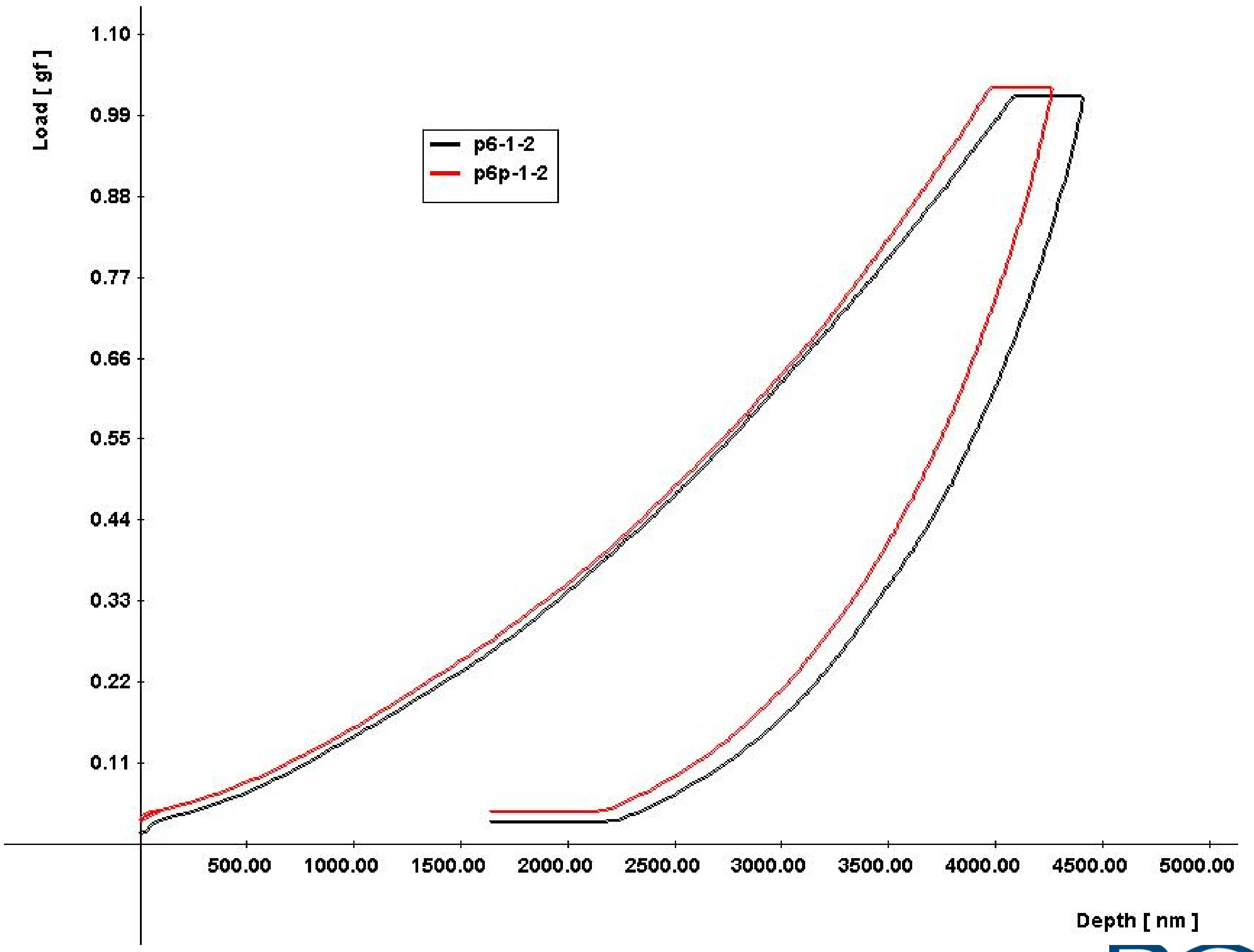

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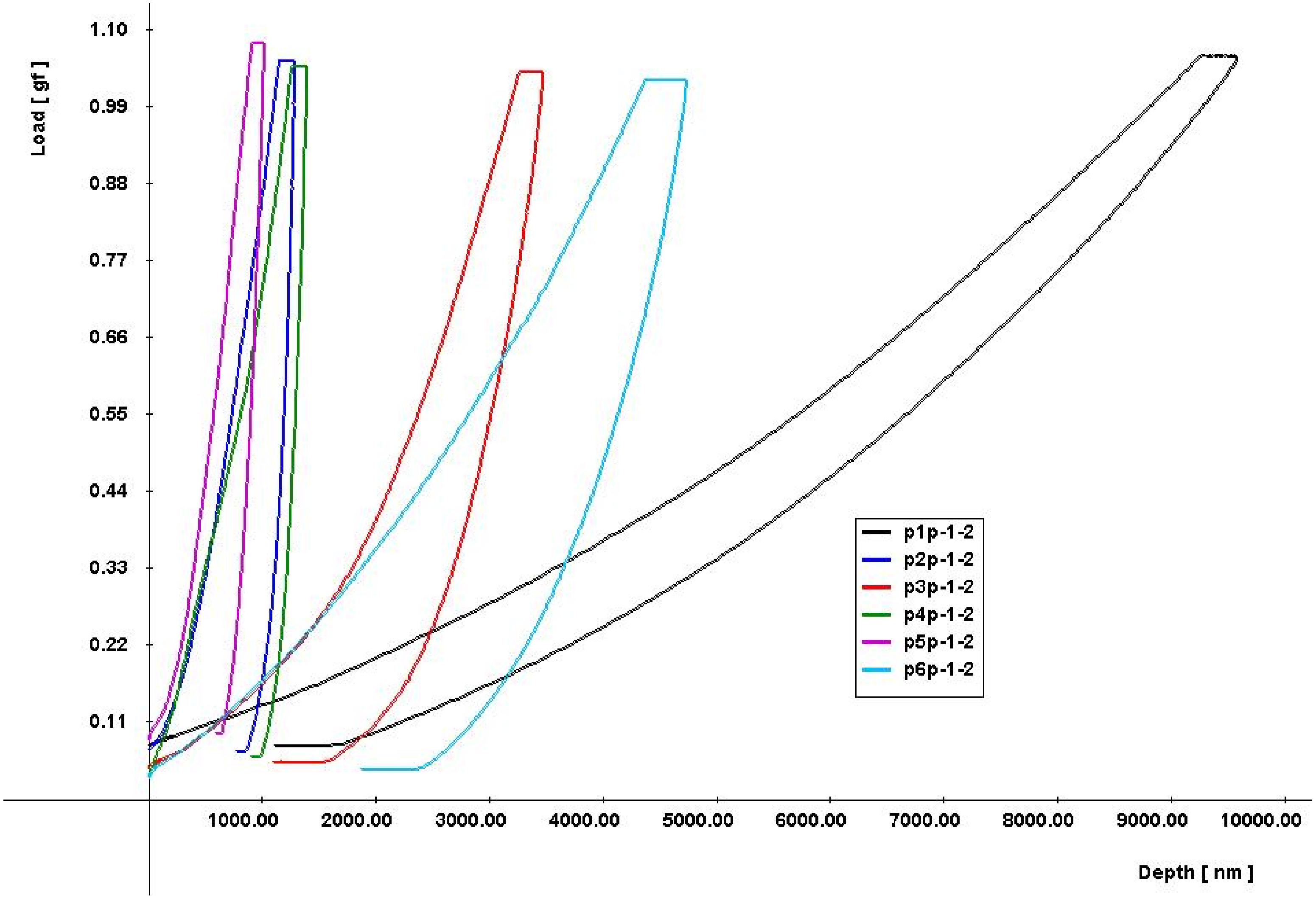

5. What We Are Doing

NEXT STEP - Benchmarking

<u>The ADVANCE PROJECT</u>
(FP7 Euratom project funded by the EU)