Die BAM
in Berlin & Brandenburg

Plasma Germany

Fachausschuss Normung/Standardisierung

Bericht auf der Herbstsitzung

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Ruhr-Universität Bochum, 14. November 2011

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Inhalt

Aktuell:
Sitzung ISO TC 229 "Nanotechnology"
Vertreter BAM: G. Reiners

Oktober 2011:
Sitzung ISO TC 172 SC 3 "Optical Coatings"
Vertreter BAM: U. Beck

Neuer Haftfestigkeitstest als Ergebnis einer Defizitanalyse zum Stand der Normung

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ISO TC 229
Nanotechnology

G. Reiners als Chair des deutschen Spiegelausschusses

- **Terminilogie "Nano"**
  JWG von ISO TC 229 & IEC 113
- **Measurement & Characterization "Nano"**
  JWG von ISO TC 229 & IEC 113
- **Measurement, Characterization and Performance Evaluation**
  WG 1 von CEN TC 352
- **Commericial and other Stakeholder Aspects**
  WG 2 von CEN TC 352
- **DIN NA 062-08-17 als Spiegelausschuss**
  von ISO TC 229 *und* CEN – TC 352 (mandatiert)

siehe Bericht letzte bzw. nächste Frühjahrssitzung

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ISO TC 172 SC 3
Optical Materials

U. Beck als ISO-Experte und Mitglied des dt. Spiegelausschusses

- **WG 1 "Raw Optical Glass"**
  P. Hartmann (Schott, D)
- **WG 2 " Coatings"**
  G. Boultbee (JDSU, USA), H.J. Niederwald (ZEISS) & U.Beck
- **WG 3 "IR-Materials"**
  T. Otaki (Nikon, JP)

Generelles: **NWI in WG 1 vs. new WG "Optical Polymers "**

Spezielles: **Internationaler Ringversuch zum "Cheese Cloth Test"**
ISO 9211-4: "mild abrasion vs. adhesion"
Fazit: riesige Streuung primär bedingt durch unterschiedliche Definition des Versagens!

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Adhesion of Coatings

Motivation

- diversity of materials (coatings/substrates)
- plenty of applications (precision optics vs. mechanical engineering)
- wide thickness range (lower nm to upper µm)
- great variety of adhesion tests, more than 100 (Mittal et. al.)
- poor comparability and reproducibility
- lack of quantitative tests (force/area, i.e. N/mm^2) except the pull-off/tear-off test for adhesion strength below bonding strength
- best case: qualitative ranking of adhesion quality
- solution to the problem: pull-off test in a centrifuge up to 100 N/mm^2

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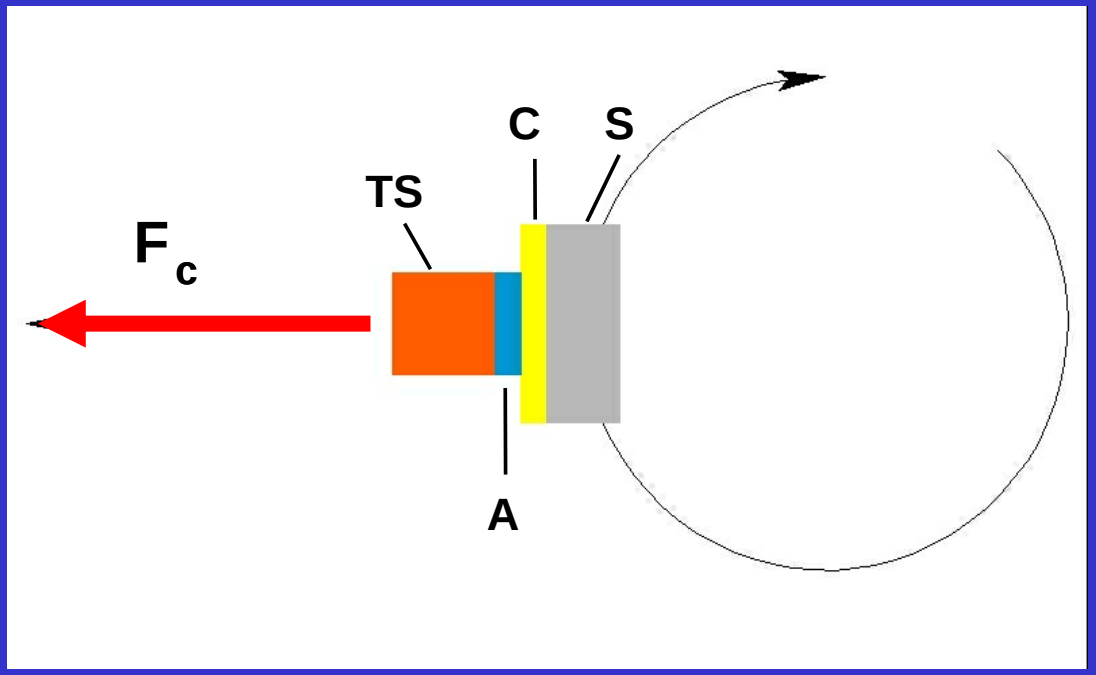
Centriguge Test

Operational Principle



Centrifugal force

$$F_C = m\,\omega^2\,r$$

Adhesion (A)/bonding (B) strength

$$\sigma_{A/B} = F_C / A$$

Test stamp (TS) glued on a coating/substrate (C+S) system at the adhesive surface (A)

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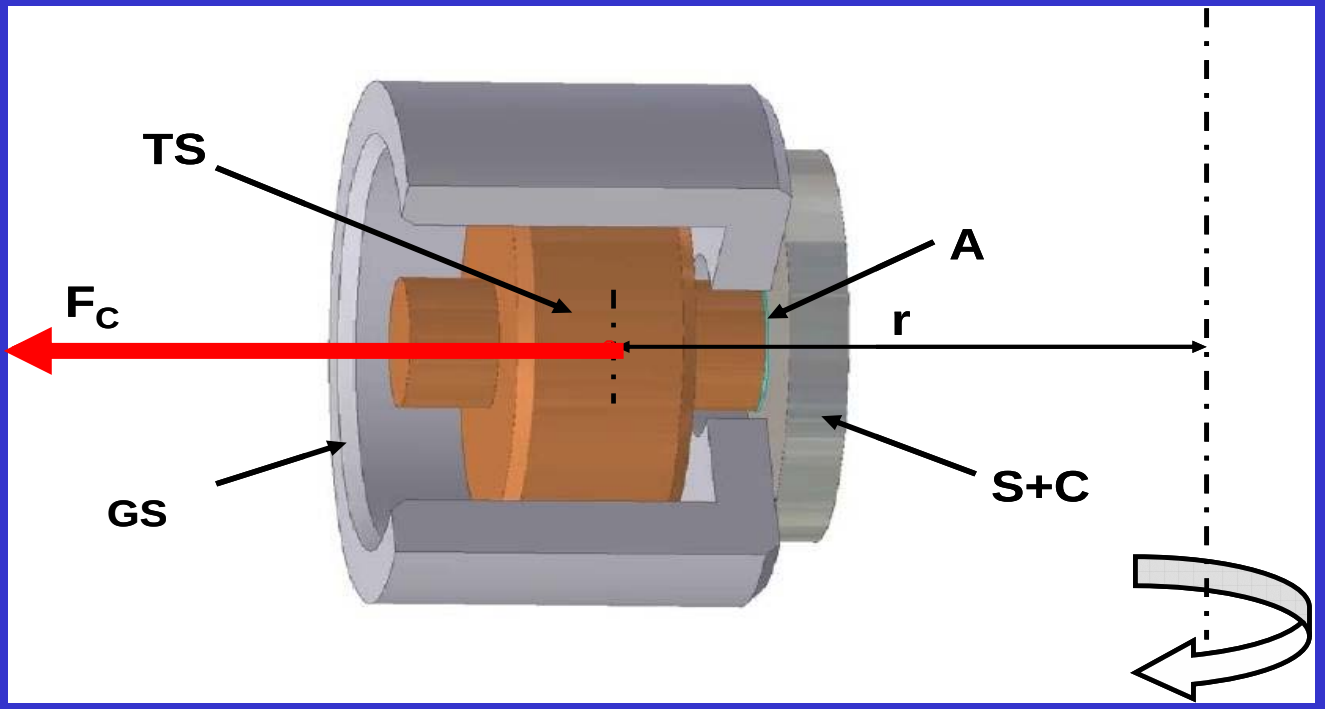
Detail of Testing Unit

Sample Support



Sample support & adhesive application kit
Guiding sleeve (GS) for test stamp (TS), adhesive surface (A) and sample consisting of coating (C) and substrate (S)

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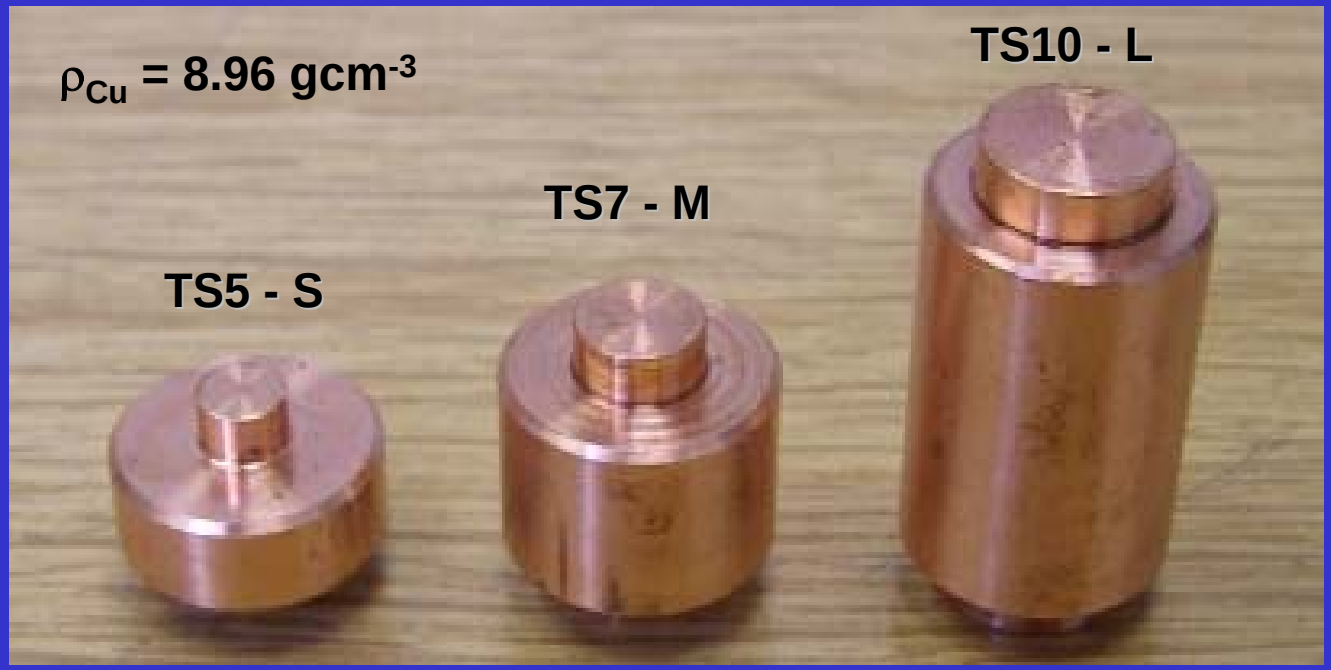
Detail of Sample Support

Test Stamps



Cu test stamps TS5-S, TS7-M and TS10-L

- **5, 7 and 10 refer to the diameter of adhesive surface in mm**
- **S, M and L refer to the shaft lengths of 6, 11 and 22 mm**
- **test stamp mass ranges from 10.4 g (TS5-S) to 38.7 g (TS10-L)**

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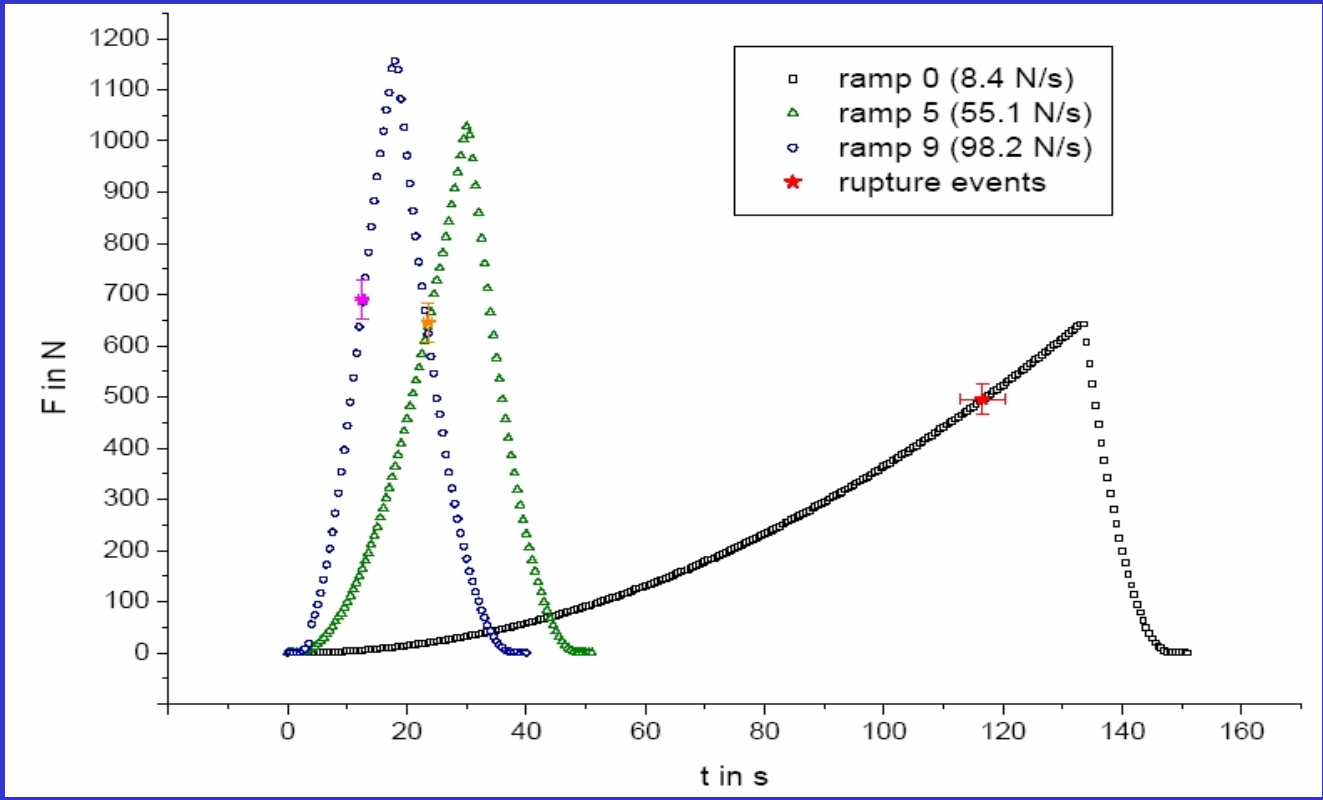
Bonding Strength

Effect of Acceleration Ramp



Force over time vs. acceleration ramp

Cu test stamp – PU – Al substrate using test stamp TS7-M (n = 12)

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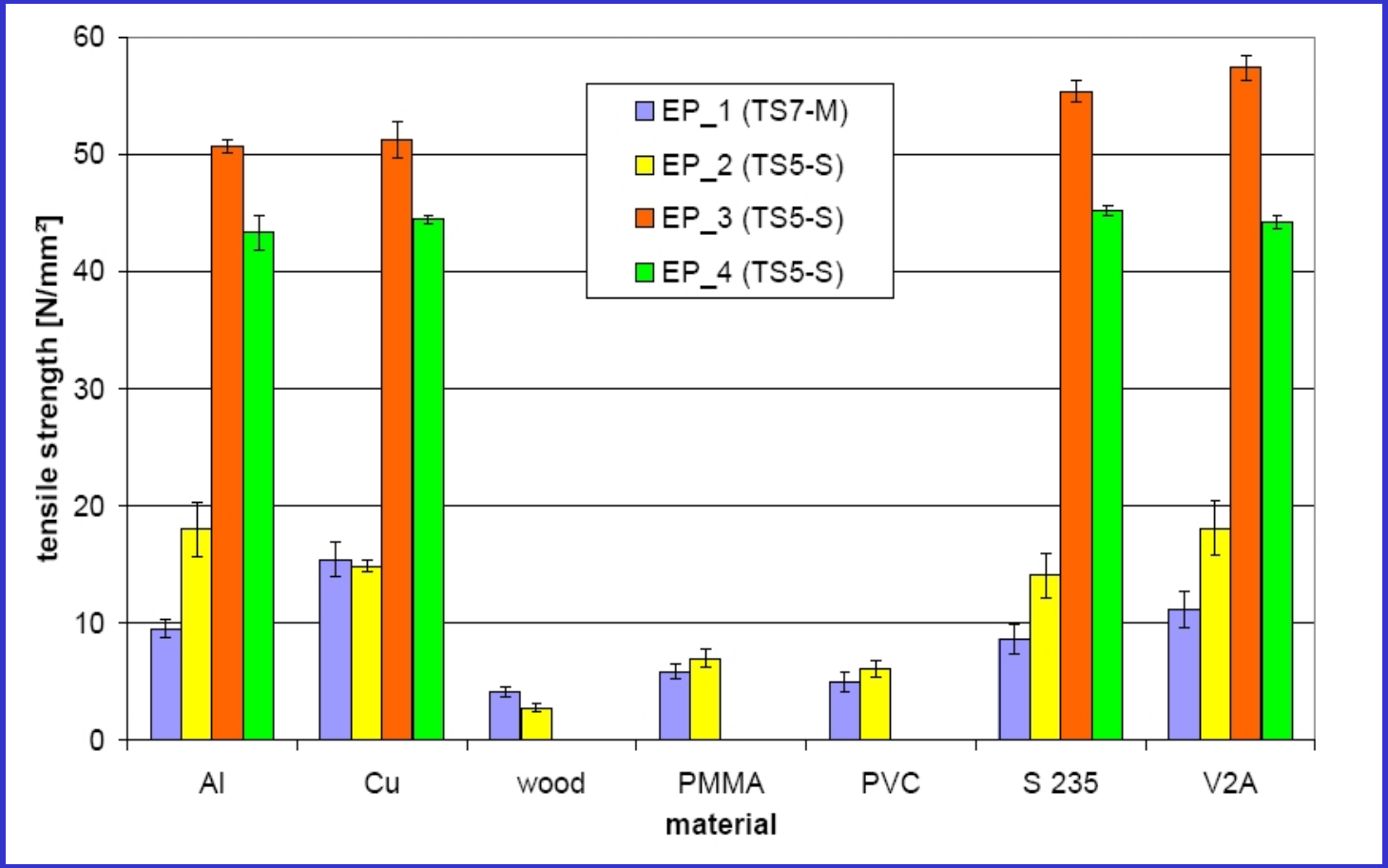
Bonding Strength

Epoxy (EP) Adhesives



Cu - EP - X
n = 12
ramp 5

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Adhesion Strength

Optical Coatings on Glass and CaF_2

Adhesion could no be discriminated by ISO 9211- 4 tests for samples 1 to 9

sample ID	substrate		adhesive layer	functional layer	protective layer
	material	thickness			
1	B270	3.4 mm		Ag / Ta	Ta_2O_5
2	B270	3.4 mm	Cr	Al	oxide layer
3	B270	3.4 mm		Ag	Ta_2O_5
4	CaF_2	2 mm		Ag	Ta_2O_5
5	CaF_2	2 mm		Ag	Ta_2O_5
6	B270	3.4 mm		Ag	Ta_2O_5
7	CaF_2	2 mm		Ag	Ta_2O_5
8	B270	2 mm	Cr	Au	oxide layer
9	B270	2 mm		layer stack	

Centrifuge test: Cu test stamp - EP - coating/substrate, TS10-L, ramp 2

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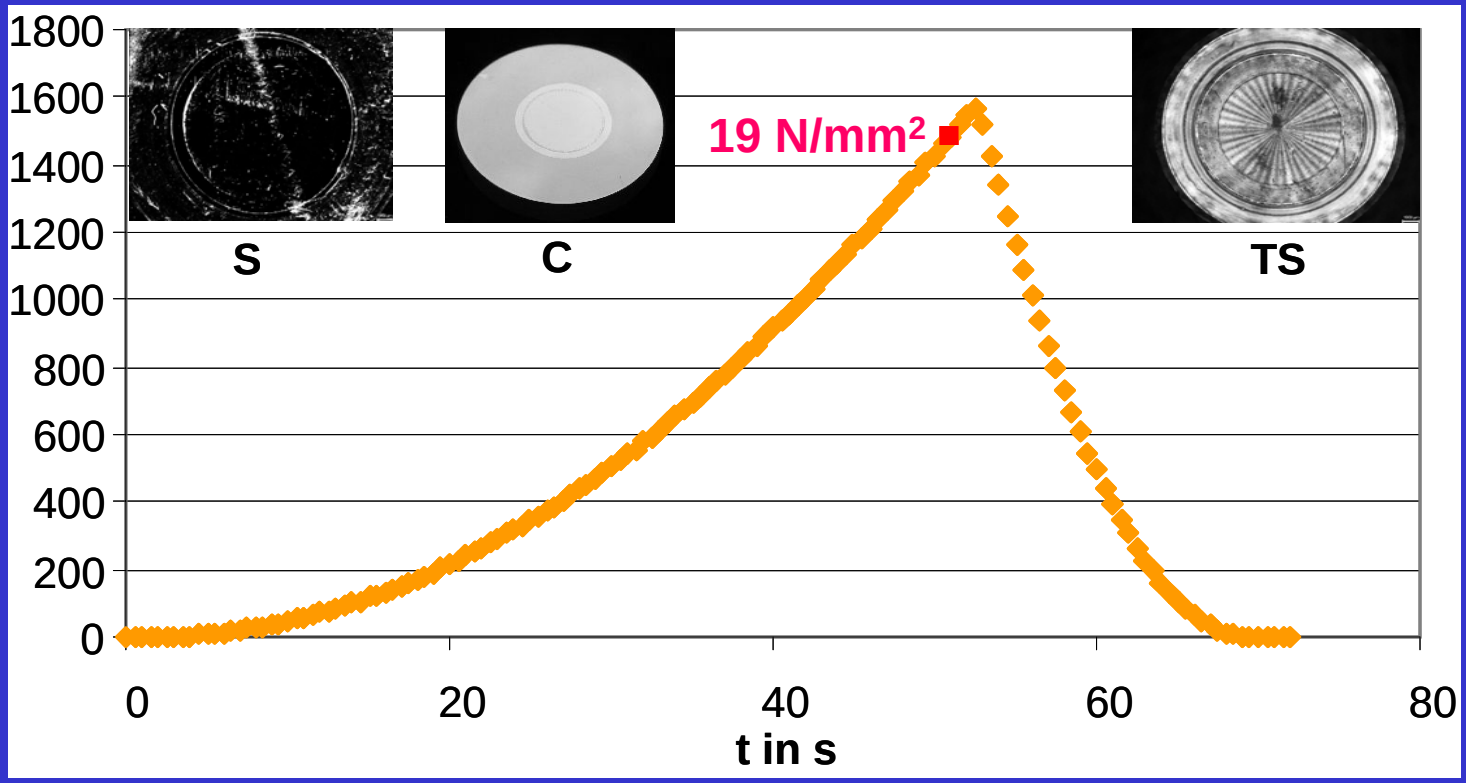
Adhesion Strength

Delamination within Layer Stack on B270



Delamination within layer stack (sample 9):
adhesive failure in between layers

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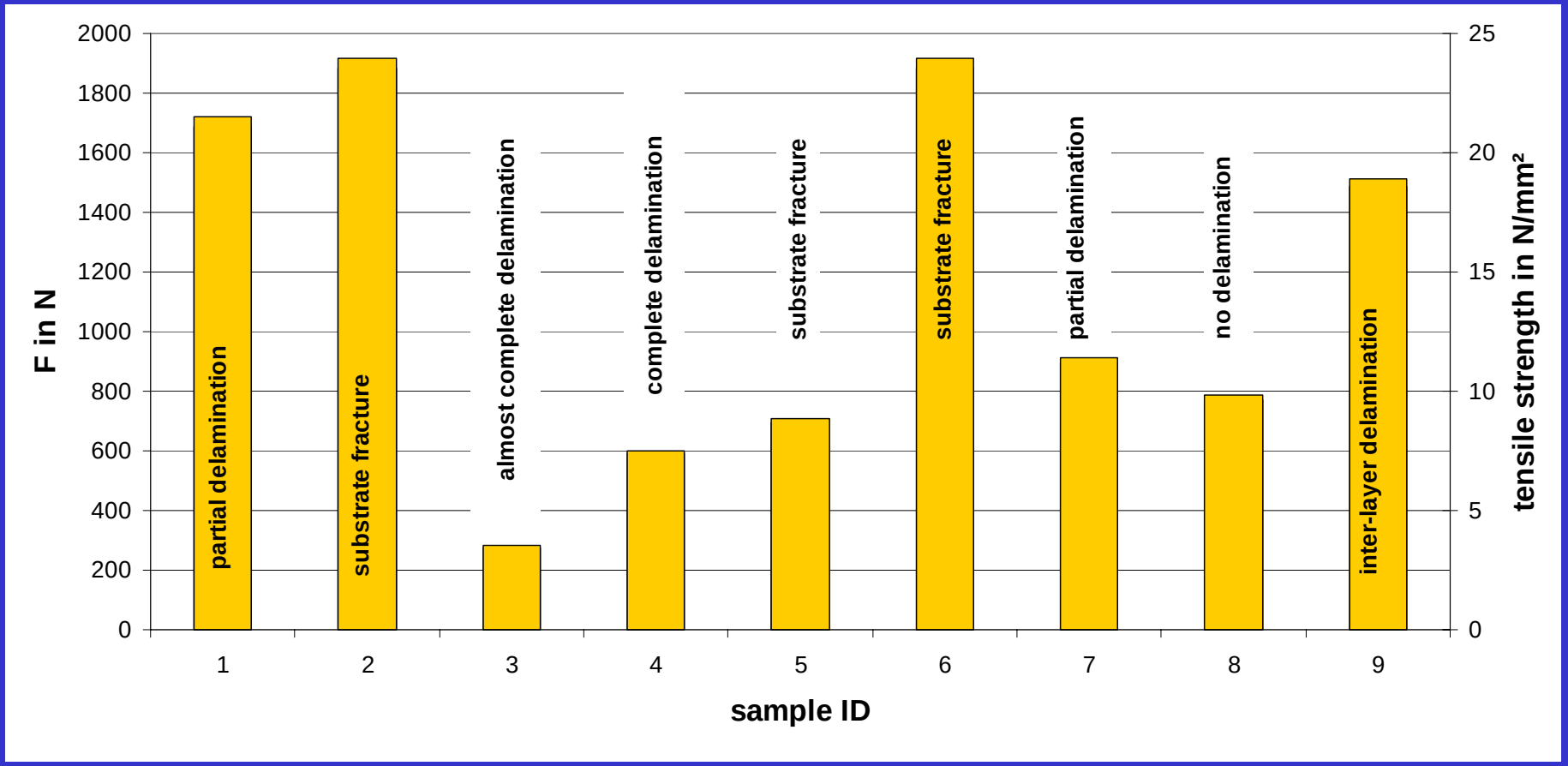
Optical Coatings

Testing Results in Comparison



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Centrifuge – Commercial System

Adhesion Analyser

Position-coded & rpm-correlated data transmission of rupture events

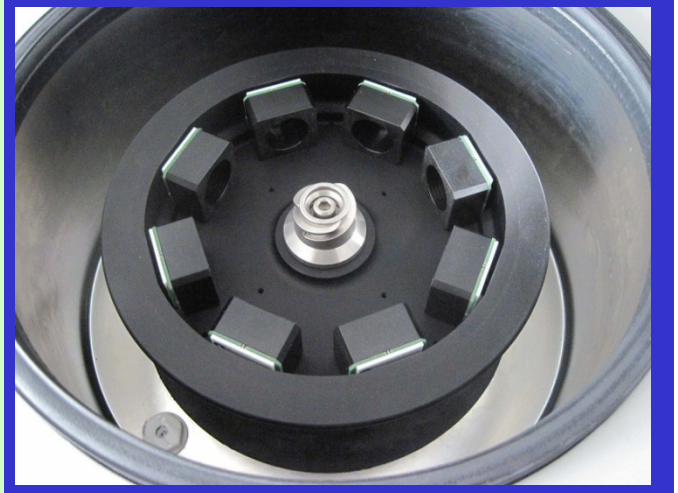
- LUMiFrac
- Rotor LFR-ST: 200.42

- 8 testing units

- ν = 13 000 min^{-1}
- a_c = 13 715 *g*
- F_{max} ≈ 4.6 kN

- E_{kin} = 10 000 Nm

www.lum-gmbh.de



Upgraded desktop centrifuge for commercial application

Finite element designed drum rotor equipped with eight testing units

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Summary

Bonding & Adhesion Strength

1. Tensile strength in absolute numbers: N/mm^2
2. Centrifuge test meets the requirements of DIN EN 15870 (DIN EN 26922)
3. Discrimination of adhesion beyond the abilities of other tests e.g. ISO 9211-4
4. Easy to use: desktop plug-and-test system, programmable test cycles (ramps, fast and slow pull test, alternating loads), single-sided sample support instead of two-sided sample clamping
5. Fast, up to eight samples simultaneously within 5 min. including mounting under identical test conditions and similar curing times of the adhesives (at least a factor of 10 faster compared to the pull-off test)
6. Defined accuracy, much better precision and comparability
7. High flexibility in terms of materials (polymers, glass, ceramics, metals); tensile tests both of bonding and adhesion strength in a wide force range from the lower N to the upper kN with the same testing equipment
8. Cost-effective and reliable, no regulatory measures on operational safety required (up to E_{kin} = 10 000 Nm)

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