Cavity Processing

- Reminder on ILC request for cavity processing to TTC community
  - Issues to be answered
  - Status before TTC-FNAL meeting
  - Load to experts in the WG1 TTC-FNAL meeting
- Industrialization of EP at DESY:
  - CARE EU investigation of EP QA methods
  - XFEL activities for EP by industry
- Latest 9-cell cavity results at DESY
  - Benefit of alcohol rinsing

Example Request to the TTC and hence to the TB

The need for further information from TTC

Nonetheless more specific details are required for the ILC R&D to compile a focused programme yielding high-gradient performance. Several institutes are pursuing these goals. Currently, the various setups result in a large variety of recipes. Although, the basic recipe for "final surface preparation" has been agreed upon (EP, HPR and 'In-situ' bakeout as described in the ILC BCD7) several other activities are not consistent between the laboratories such as after-EP rinses, rinse times etc.

A significant effort has now been directed towards high gradient work on the basis of the documents mentioned above. The S1 Task Force is seeking advice on the following issues to improve the yield of the "final preparation steps" :

- Optimum cavity preparation process
  - A detailed list of preparation steps would be desirable.
- Optimum set of EP parameters established today
- Optimum set of HPR parameters
  - A proposal on how to implement a consistent and verifiable parameter set for these systems would be desirable
- Optimum set of bakeout
  - An optimum parameter set should include temperature, duration and vacuum.
- List of critical process parameters to be monitored during cavity preparation
  - This applies to all of the processes above
  - Recommended monitoring devices for process control

The task force would like to request a document prepared by TTC which includes the aforementioned information. This document should serve as a guide book/manual. It is assumed that the upcoming TTC Meeting at KEK will address this with a focus on a next generation EP systems for production. The task force hopes that the resulting document will help to synchronize the efforts on the cavity preparation

D.Proch, LCWS 2007

Prep. official answer

- extract the essence from existing material
- produce a well structured document
- add background information completing the picture

TTC-FNAL Meeting, WG2

Conclusions:

- At Frascati TTC meeting we identified many new rinsing / cleaning methods after EP to reduce field emission – list generated

- At KEK TTC meeting we focused efforts on two areas, one QA of electrolyte and second was basic study reducing spread in performance (better rinsing methods)

- At FNAL TTC meeting – results from both rinsing methods and better QA were presented

  Now a better understanding is emerging

Summary documents of EP working groups at the TTC meetings Frascati

http://ilcagenda.cern.ch/conferenceDisplay.py?confId=desya0561&view=cdsagenda&showDate=all&showSession=all&detailLevel=contribution

and KEK

https://indico.desy.de/conferenceDisplay.py?confId=92

Compiled by D.Proch

Content:

Example page of Higo`s data collection

Chemical surface treatment process									
		units	12/5/05 Decision	DESY	Jlab	KEK (Nomura)	KEK (STF)	FNAL SMTF	ANL
Electropolishing				EP 1/9	EP 1/5	EP	EP 1/9		
	Situation			Horiz	Horiz	Horiz	Horiz		filled
	Control			const voltage		const current	continuous		
	Voltage	V		17?		15-25	const current		15 20
	mode			continuous	continuous	continuous	15-25		pulsed
	Current	mA / cm^2		49-58		40~50	40-50		30 to 50
	Power	W / cm^2		1					
	Temperature of liquid (start-end)	C		24-35		25~32	25-32		28 32
	Temperature of cavity	C				30~35	30-35		
	Acid flow rate	l/min		9-11		6	6		agitate
	rotation speed	rpm		1.5-2	1	1,4	1,4		
	H2 screen details			360 degrees	180 degrees	180 degrees	180 degrees		
	nitrogen purge for H2			30lpm	1cfm	none	none		
	cathode masking			iris/BT	iris/BT	iris/BT	BT		
	seal material			PVDF		viton	viton		

Example page of Higo`s data collection, cont.

	Tolerable Nb content	gpl		10		9	9
	Heat exchanger			PVDF tube	PVDF tube	PFA tube	PFA tube
	Acid quality (grade)			electronic	electronic	reagent	reagent
	storage volume	l		150	240	1000/100	1000
	masking material			PTFE tape	PTFE tape	PFA film	PFA film
	cathode to NB ratio						
	Cathode material	AL purity		100		1100 series	1100 series
	Cathode shape			tube	tube	tube	tube
	Cathode dia			1.0 inch	1.3inchs	25 mm	25,0
	etching speed	micron / min		0,4		0,4	0,4
	total removal	micron		180-200		80	80
	additional removal	micron		?		30	30
	total time for a treatment	hr		6+2 h		3.5	3.5
	contact location			2 stif ring, 2 dish head		all EQ plus beam tube	all EQ plus beam tube
	experience			> 200hr		> 3600 hr	0
	diff temp in to out	C		8		10	

TTC-FNAL Meeting, WG2 summary

- The summaries define an extended parameter space for EP, Rinsing, Drying, Clean Water, Clean Assembly, Heat treatment.. and other procedures that are known to give good cavity results.
- There are detailed Tables at the end
- Some furthur work is needed to add information from more recent studies, e.g Henkel.
- An executive summary is needed.
- S0 Task Force should use this extensive “data base” to define a uniform preparation procedure for the three labs to follow.

TTC will publish and update this compilation of information, could evolve into kind of handbook for cavity treatment to serve the whole TTC community D.Proch, LCWS 2007

The structured answer as proposed by TB

incl. suggested authors, partly tbc(onvinced)

1.	Optimum cavity preparation process	H.Padamsee, K.Saito
	general overview based on Sect. 2-4; incl. assembly techniques	
2.	Optimum set of EP parameters for 9-cell cavities as established today	J. Mammosser
2.1	Recommended EP parameter	J. Mammosser
2.2	Recommended acid quality monitoring	A. Matheisen
2.3	Recommended rinsing parameter	J. Mammosser
	Supporting material	
	Comparison EP parameter	T. Higo
	Status of acid quality monitoring	A. Matheisen
	Results rinsing studies; list of possible rinsing methods and current status	
	HF rinsing / short EP	K. Saito
	Ultrasound degrease	J. Mammosser
	Alcohol	A. Matheisen
	H2O2	E. Kako
3.	Optimum set of HPR parameter	P. Kneisel
3.1	Recommended HPR parameter	P. Kneisel
3.2	Recommendations wrt process quality monitoring,	
	e.g. force, particle count	D. Reschke / P. Michelato
	Supporting material	
	Comparison of HPR systems	P. Michelato
	water quality	Rothgeb /Saeki
4.	Optimum set of bakeout parameter	
4.1	Recommend bakeout parameter	B. Visentin
	Supporting material	
	Comparison of bakeout procedures	Visentin / Ciovati / Furuta

Industrial Study to define QA method for EP Electrolyte

Supported by the EU CARE Project

HENKEL
Lohnpoliertechnik GmbH & Co. KG
An der Autobahn 12, D-19306 Neustadt-Glewe
Tel.: 0049(0)38757/66-0* / Fax: -122
Email: info@henkel-epol.com

Abschlussbericht

Studie zur Untersuchung des H2SO4/HF-Elektrolyten zum Elektropolieren von Niob

zwischen dem

Deutschen Elektronen-Synchrotron DESY
Notkestr. 85
D-22607 Hamburg

und der

Henkel Lohnpoliertechnik GmbH
An der Autobahn 12
D-19306 Neustadt-Glewe

This study consists of detailed descriptions of
-possible QA methods (data bank, presentations, meetings)
-availability of measurements by commercial companies
-first QA measurements on new/old EP electrolyte
-proposal for detailed calibration studies with selected QA methods

List of EP Elektrolyte QA methods

Spektroskopische Verfahren

1.1 Molekularspektroskopische Verfahren
- Ultraviolett(UV/VIS)-Spektroskopie
- Infrarot-Spektroskopie (FT-IR / ATR) (Fourier transformation infrared spectroscopy –attenuated total reflection)
- Raman-Spektroskopie
- Kernmagnetische Resonanz Spektroskopie (NMR)
- Elektronen-Spin Resonanz-Spektroskopie (ESR)
- Massenspektrometrie (MS)
- **Massenspektrometrie mit induktiv gekoppeltem Plasma(ICP-MS)**
  Optical Emissions-Spektr. ICP-OES

1.2 Atomspektroskopische Verfahren
- Atomabsorptions-Spektrometrie(AAS)
- Atomemissions-Spektrometrie (AES)
- Emissions Spektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)
- Röntgenfluoreszensanalyse (RFA)

(FT)IR (Fourier transformation infrared) Spectroscopy

Principle: Wave lenghts dependent stimulation of bonds between atoms for mechanical oscillation through absorption of light within the IR-range (wave lenghts of approx. 0,8-500 µm). The hereby obtained interferogrammes contain the complete radiation absorption of the trial after wave length and intensity as Fourier sum of all spectral lines. Resultant is a better dissolving ability as with the classic IR-spectroscopy.

\+ Direct measurement of properties of the electrolyte
Qualitative and quantitative analyses are possible

\- Main problem : electrolyte resistant glas materials for cuvettes must be found (and all the same transparent in the IR-range)

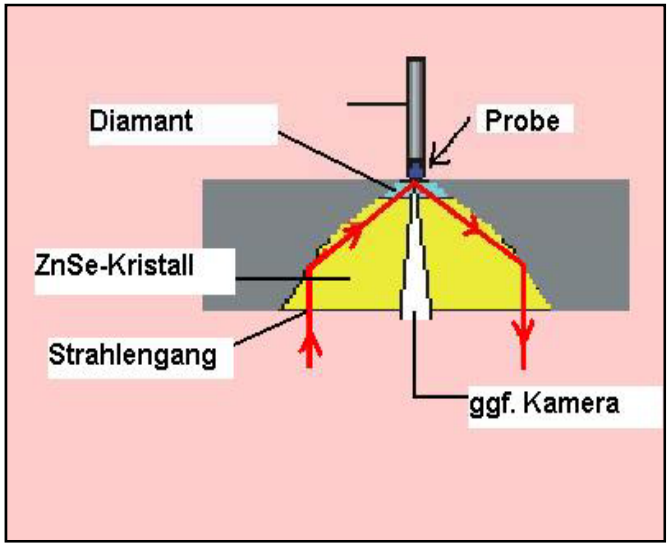
Costs: >15.000,-€

Producer: Varian, Brukeroptics, Shimadzu, Thermo,...

HENKEL Lohnpoliertechnik GmbH An der Autobahn 12 / D - 19306 Neustadt-Glewe (M-V)
Tel. +49 (0) 38757 66 0 / Fax. +49 (0) 38757 66 122 / Email : info@henkel-epol.com / www.henkel-epol.com

ATR-Probe (attenuated total reflection)

HiTec Zang ATR-Probe used by RWTH Aachen



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ICP-OES (optical emission spectroscopy)

Principle: the testsubstance is ionised in an argon plasma (6000K–12000K), the effect is an optical emission you can analyse in a spectrometer

\+ simultaneous multielement analyse upto 70 elements (qualitative und quantitative)

better repeatability than AAS because insensitivity of interferences

\- no information about molecular characteristics

costs: approx. 60.000,-€

producer: PerkinElmer, Varian, Spectro, Thermo...

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Tel. +49 (0) 38757 66 0 / Fax. +49 (0) 38757 66 122 / Email : info@henkel-epol.com / www.henkel-epol.com

List of EP Elektrolyte QA methods, cont.

Chromatographie
- **Gaschromatographie (GC)**
- **Flüssigkeitschromatographie(HP-LC)**
- **Ionenchromatographie (IC)**
- **Kapillarelektrophorese (CE)**
- **Isotachophorese (ITP) (Separation of Ions by electric fields)**

(Elektro)chemische Nachweismethoden
- **Ionensensitive/selektive Elektroden (ISE)**
- **pH-Elektroden**
- **Karl-Fischer-Titration zur Wassergehaltsbestimmung**
- **Titrationen(Volumetrie)**

Messung physikalischer Eigenschaften
- **Dichtebestimmung**
- **Leitfähigkeitsmessung**
- **Brechzahlbestimmung**

Summary of Industrial Study to define QA method for EP Electrolyte

- In total 23 methods for analyzing the EP electrolyte quality have been examined:
  - Molecular-Spectroscopy
  - Atom- spectroscopy
  - Chromatography
  - Electro-chemical methods
  - Physical methods
- Three most promising / practical methods will be qualified (calibrated) in a second industrial study
  - **FT-IR / ATR**; **ICP-OES**; **ITP**
- So far the study is in German language
  - But could be translated

- **Report will be available by TTC under the rules of FP6 EU R&D program**

Industrialization of electro polishing at DESY in preparation of the XFEL project

Status Report by
Axel Matheisen
DESY Hamburg

One major surface preparation technique for the XEL is the electropolishing of the niobium cavities

Actually only the prototype ep facility for nine cell cavities is operational in Europe at DESY

To study difficulties of industrialization and to get cost estimates in an early state a study on industrialization of electropolishing was launched

Content of Study

EP application basing on the experiences at DESY

Main EP removal of damage layer (150-180 µm)
- Clean environment necessary
- No HPR or class 10 areas needed
- use of ultra pure water for rinsing recommended
- No special transportation kit necessary

Fine EP removal of 20-40 µm Niobium in preparation for test
- HPR and Ultra pure water necessary
- Class 10 cleanroom for assembly and leak test necessary
- Trained personal for assembly necessary
- Special transportation kits needed

The study is limited to main EP due to high investments necessary at the companies to fulfill requirements on fine EP

DESY	Delivery of cavities mounted in frames and transport flanges
Company	Performing of main EP (160-180 µm surface removal) Delivery of log files and protocols of the process to DESY Rinsing of cavity to >12 Mohm cm resistivity Installation of transport flanges in clean environment no intermediate drying of cavity allowed during flange assembly Transport of cavities filled with ultra pure water to DESY
DESY	Outside TI treatment 800 C H2 degassing fine EP / flash BCP and assembly for test HPR test

According to European rules a limited call for tender was launched

5 companies singed in to get the specification and study information's

2 Companies rejected because of
difficulties to get legal permission to use EP mixture containing HF acid
2 companies placed an offer according to EU rules
1 Company placed the offer delayed. The offer had to be rejected.

Orders were placed to 2 companies offering different solution

Status

Contract placed for January 07

- 15 Cavity treatments for new cavities at Accel Instruments
- 15 Cavity treatments for new cavities at Henkel Lohn Elektropolitur

Schedule

- Set up of facility and acceptance of hardware by DESY Mid 07
- Commissioning of infrastructure with cavity delivered by DESY
- Qualification of infrastructure and processes
  with a dummy cavity supplied by DESY
- Delivery of cavity main EP treatments from August to October 07

TTC-FNAL Meeting, WG2

Conclusions:

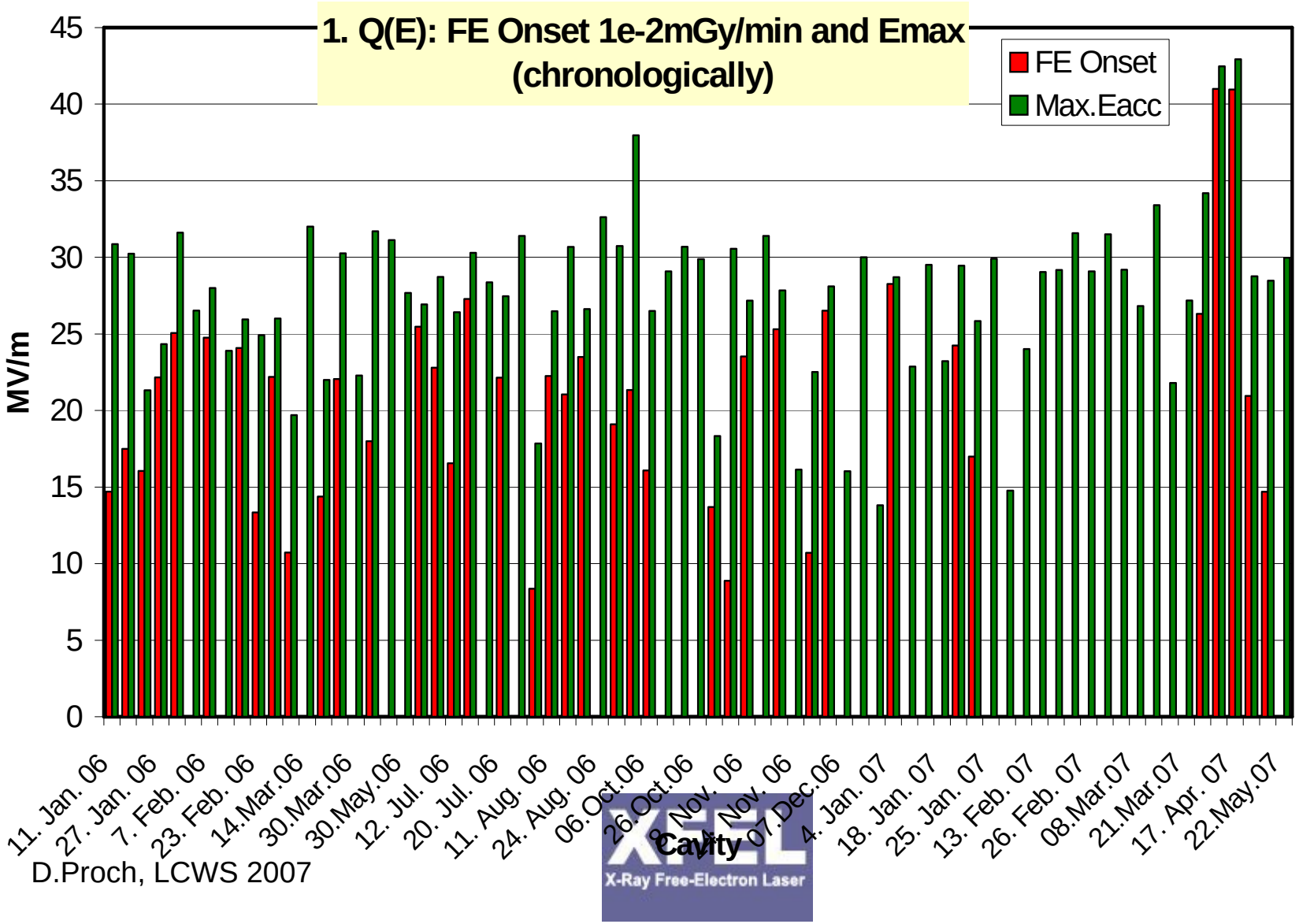
- Control of field emission being addressed differently at each lab
  - Degreasing at JLab
  - BCP+Alcohol rinse at DESY
  - Fresh Acid +Ozone water rinse at KEK
  - Limited success so far
    - Field emission has been reduced (JLab above 30MV/m) but not eliminated
    - There is a possibility that even small amounts of x-rays could be the cause of the early quenches?

Need a consistent approach to addressing field emission and currently not one single method has enough evidence to support global implementation
This should be our highest priority!!

Development of Field Emission since Jan 06

compiled by D.Reschke

- Analysis of 1. Q(E)-results of all nine-cell tests (NOT preparations):



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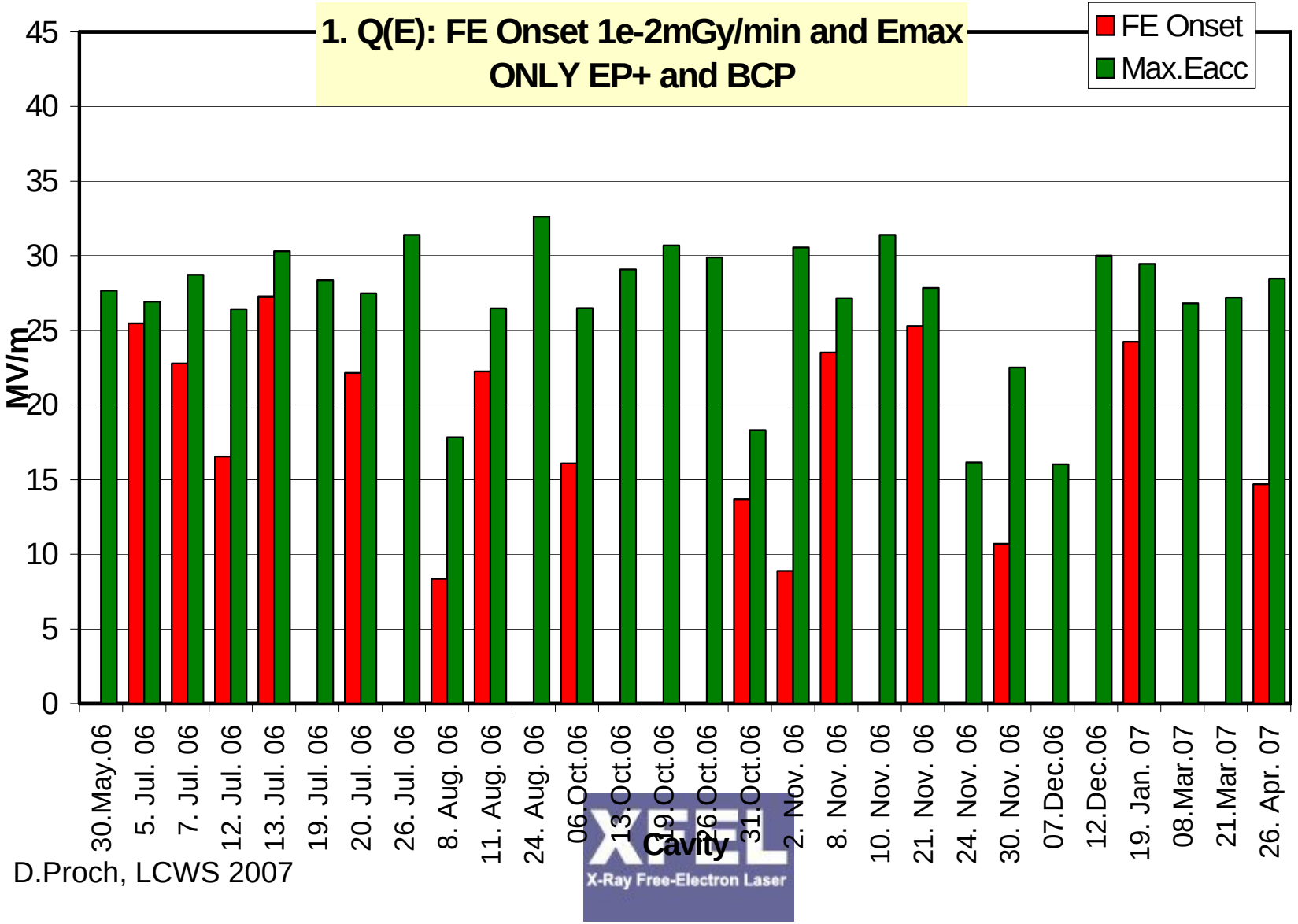
X-Ray Free-Electron Laser

Development of Field Emission since Jan 06

compiled by D.Reschke

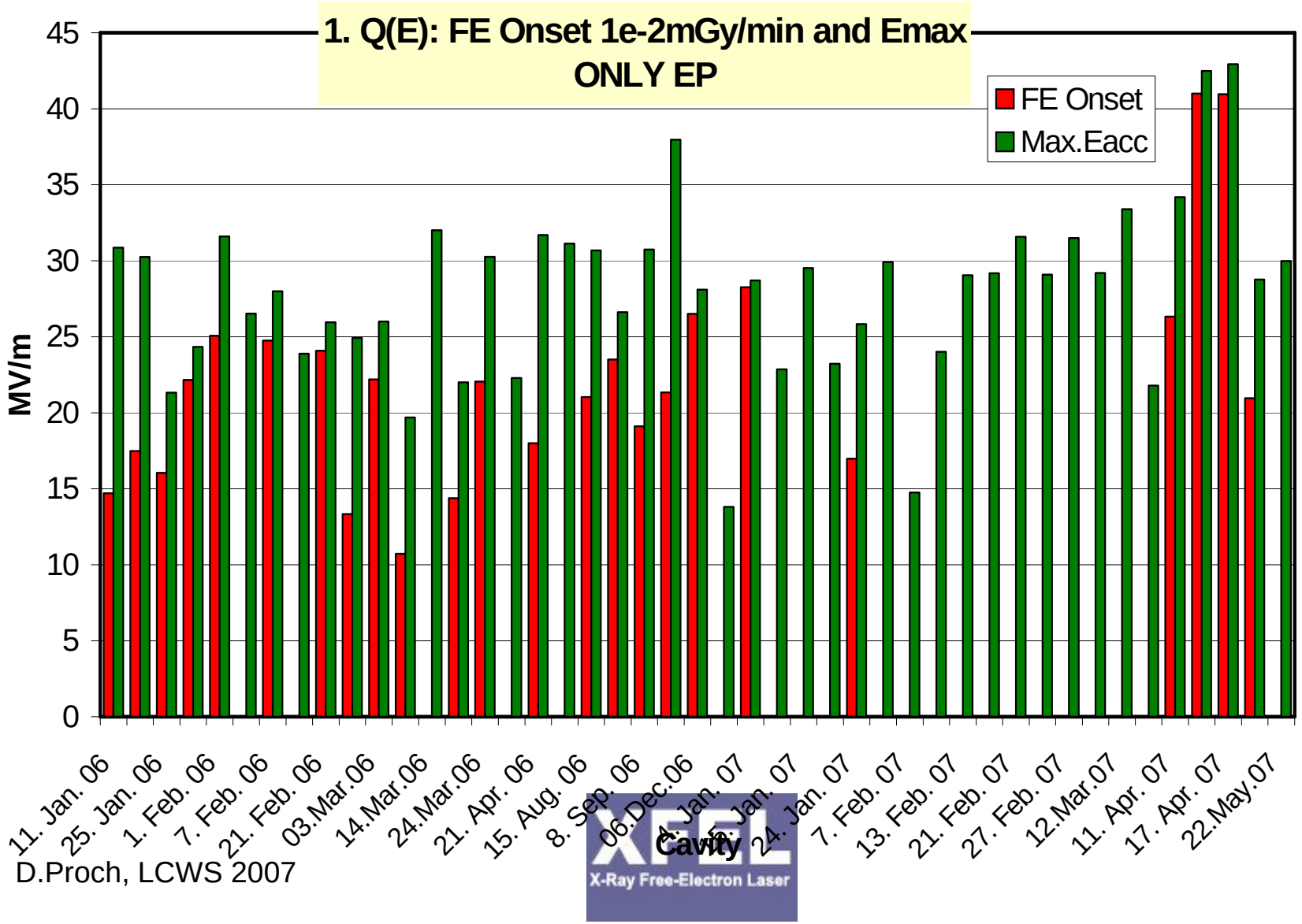
- Analysis of 1. Q(E)-results **only BCP / EP+ cavities** (all tests, not preps):



Development of Field Emission since Jan 06

compiled by D.Reschke

- Analysis of 1. Q(E)-results **only EP cavities** (all tests, not preparations):



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X-Ray Free-Electron Laser

Emissions Spektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)

Prinzip: Probensubstanz wird mittels eines Argonplasmas bis hin zur Ionisation angeregt, sodass es zur optischen Emission kommt. Das Spektrum wird dann analysiert.
Vorteil: simultane Multielementanalyse bis 70 Elemente (qualitativ und quantitativ) gleiche Nachweisgrenze wie AAS aber bessere Reproduzierbarkeit als AAS, da unempfindlicher gegen Anregungsstörungen.
Nachteil: keine Aussagen über Bindungszustände / Moleküleigenschaften (da „atomisiert“)
Kosten Gerät: ca. 60.000,-€
Anbieter: Varian, Spectro, PerkinElmer
Analysedauer: 3 Min. pro Analyse für 10 Elemente
Analysekosten: 128,-€ pro Stunde Labor
Analyseergebnis: Elementanalyse: Es wurden in beiden Proben (kalt und warm) nur sehr geringe Elementmengen gemessen, nur Eisen (Fe) wurde in beiden Proben festgestellt, dabei in den Warmproben ein höherer Wert als in den Kaltproben, eventuell entstanden bei der Elektrolytherstellung durch Flugrost vom Stativ. Um Verunreinigungen oder Niobgehalte festzustellen ist das Verfahren sehr gut geeignet.

Infrarot Spektroskopie (FTIR-ATR)

Prinzip: Totalreflexion an Grenzflächen wird ausgenutzt. Bei der Totalreflexion dringt die Strahlung etwas in das angrenzende Medium ein. Je nachdem, ob die Probe Strahlung absorbiert oder nicht, wird damit die Intensität des reflektierten Strahls beeinflusst. Es resultiert ein dem Transmissionsspektrum ähnliches Reflektionsspektrum

Vorteil: direkte Messung von Eigenschaften des Elektrolyten, Diamant resistent gegen aggressive Medien, Gerät ist klein und transportabel.

Nachteil: Kontakt zwischen Elektrolyt und Diamanteinfassung muss vermieden werden

Kosten Gerät: nur ATR-Einheit: 15.000,-€ bis 30.000,-€, Basisgerät ca. 80.000,-€

Anbieter: HiTec Zang, C3

Analysedauer: 3 Min. pro Analyse

Analysekosten: 500,-€ pro Woche

Analyseergebnis: Ionenmessung: Ein Unterschied zwischen dem Kurvenverlauf von Kalt- und Warmprobe konnte nicht festgestellt werden, dennoch gab es bei der Messung weiterer Proben abweichende Kurvenverläufe. Um genaueres über die Kurvenverläufe sagen zu können, bedarf es weiterer Analyseschritte mit verunreinigten Elektrolyten bzw. anderen Mischungsverhältnissen.

Isotachophorese (ITP)

Prinzip: Die ITP ist eine elektrophoretische Trennmethode, bei der Ionen und ionisierbare Komponenten aufgrund ihrer Mobilitäten im elektrischen Feld getrennt und detektiert werden Im Unterschied zur CE **(Kapillarelektrophorese)** arbeitet die ITP mit einem diskontinuierlichen Elektrolytsystem, bestehend aus einem Leit- (Leading electrolyte, Ld) und einem Folgeelektrolyten (Terminating elektrolyte, Tm)
Vorteil: keine mobile Phase notwendig, Kapillaren und Rohrleitungen aus inertem Material (FEP und PTFE) geringe Spannungen notwendig
Nachteil: Nur Ionenkonzentration messbar, es muss stark verdünnt werden.
Kosten Gerät: 25.000,- € bis 30.000,-€
Anbieter: JH-Analytik
Analysedauer: 20 Min. pro Analyse
Analysekosten: 760,-€ Methodenerstellung, erste Probe 295,-€ jede weitere 68,-€
Analyseergebnis: Ionen: Messung von 3 verschiedenen Ionen, Fluorid und Sulfat konnten eindeutig zugeordnet werden, als das 3. Ion wird Fluorsulfonsäure vermutet. Es sind bei Warm- und Kaltproben verschiedene Konzentrationen der Fluorsulfonsäure festzustellen. Je wärmer die Säuremischung hergestellt wurde desto scheinbar weniger ist von diesem Ion vorhanden. Beide Verfahren CE und ITP ermöglichen die Analyse der Ionen in der Säuremischung, aufgrund der starken Veränderung der Probenmatrix bei der CE (Neutralisation) ist die ITP die Methode die vertieft werden sollte.

ATR-Probe (attenuated total reflection)

Principle: Total reflectance at the interfaces is used. At total reflectance the radiation intrudes a little into the surrounding medium. Depending on whether or not the trial absorbes radiation the intensity of the reflected radiancy is therewith influenced. The result is a transmission spectrum similar to the reflection spectrum.

\+ Direct measurement of properties of the electrolyte
Diamond resistant against aggressive media,
Mobil

\- Contact between electrolyte and diamond mounting must be prevented (Teflonseals ?)

Costs: only ATR-unit : 10.000-15.000,-€

Provider: HiTec Zang, C3

HENKEL Lohnpoliertechnik GmbH An der Autobahn 12 / D - 19306 Neustadt-Glewe (M-V)
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