11-13 September / Eylül 2014
TÜYAP Fair, Convention & Congress Center, İstanbul
7th International Ankiros Foundry Congress
7. Uluslararası Ankiros Döküm Kongresi

«Carbon In Cast Iron, New Method For Optical Emission Spectroscopy»

«Pik Dökümde Karbon - Optik Emisyon Spektroskopisinde Yeni Metot»

Kay Toedter
H. Gerd Joosten
M Kettenis
(Spectro, BES Müh.)

6.Oturum: Süreçler ve Kontrol

6th Session: Process and Control

Oturum Başkanı/Session Chairman: Dr. Önder Orhaner (Akdaş Döküm San. Tic. A.Ş.)

Carbon in Cast Iron

the new method for improved determination of Carbon in Cast Iron

7th International Ankiros Foundry Congress

Presenter: Kay Toedter
Product Manager SMA at SPECTRO A.I.

Agenda

- General information
- Abstract
- Sample taking and related effects
- Objectives
- Definition of the analytical problem
- New approach described in 5 steps
- Results
- Resume & Benefits

General information

Total cast iron production ww:

Laminar cast iron (GJL – GGL) appr. 60%-65%.

Globular cast iron (GJS – GGG) appr. 35% increasing.

Foundries in the direct supply chain to the automotive industry have already established very effective quality control processes.

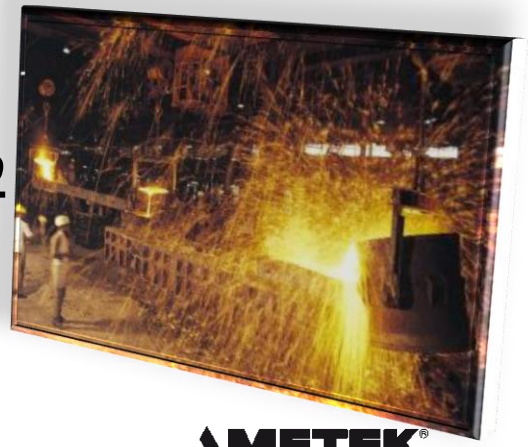
Foundries in second or third lane may have the need to increase their knowledge regarding sample taking and sample preparation.

Abstract

Carbon is the most important element in Cast Iron. It therefore has to be monitored constantly during the **melting** process, as part of the chemical composition of the melt!

Sample taking is critical, too slowly chilling produces graphite inclusions. However, it is well known, that spark emission spectrometers need **completely graphite-free** samples to produce accurate results.

This is a problem in real life and the reason to develop a new carbon determination method.



Sample Taking and related effects

All methods for total carbon detection are based on proper sample taking.

- For the combustion procedures this means to care for sufficiently raw chips, if the sample contains globular graphite. Otherwise a loss of graphite inclusions is related.

- Spark emission spectrometry (S-OES) requires a graphite free sample. This can be achieved by chilling relatively thin sample disks in a heavy copper mold.

Sample Taking and related effects

Effects of bad samples with respect to C-Determination :

- **Laminar cast iron (GJL, before GGL):**
  Samples partial grey: C determined too high. Extension of pre-burn time (e.g. to 60s) solves the problem

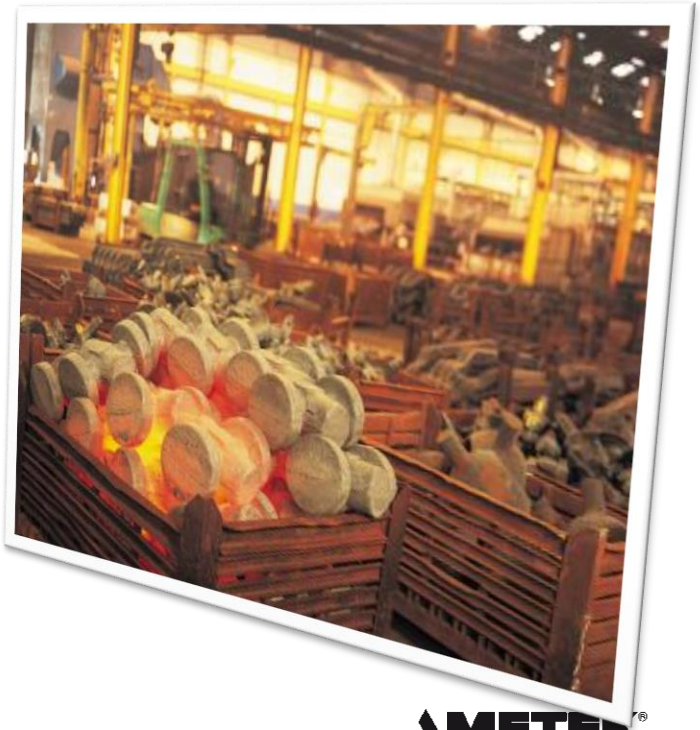
- **Globular cast iron (GJS, before GGG):**
  Samples partial grey: C determined too low.
  Extremely slow chilling (big graphite inclusions) might lead to super-elevated C-values

Objectives

- Detection of bad samples
- Enabling analysis of suboptimal samples

We will concentrate on the

most difficult case: GJS!



Definition of the analytical problem

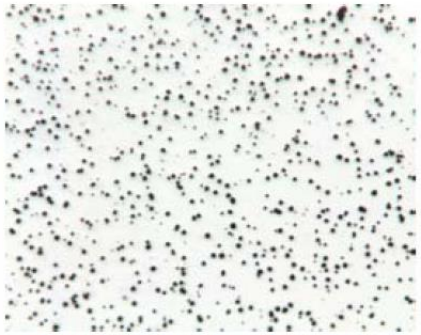
***C by S-OES*:** **3.07%**

***True value*:** **3.63%**

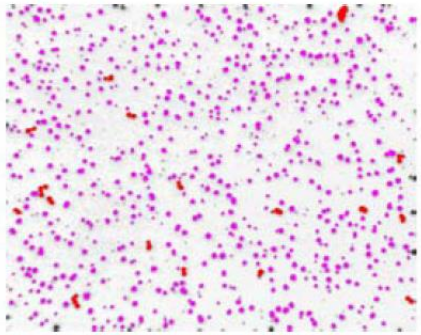
Size classes according to EN-ISO 945

	Min. µm.	Max. µm.	Avg. µm.
1		>1000	>1000
2	500	1000	750
3	250	500	375
4	120	250	185
5	60	120	90
6	30	60	45
7	15	30	22.5
8	<15		<15

Structure analysis according to EN ISO 945



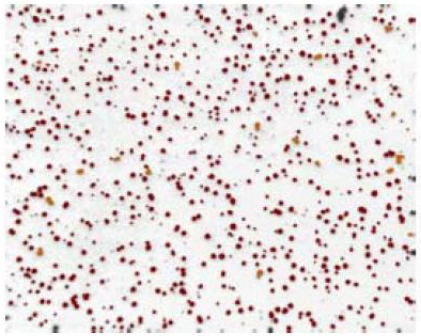
Graphite structure: Not etched 100X



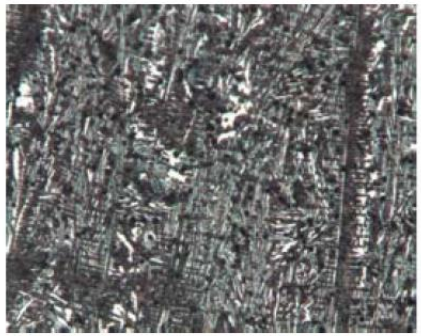
Graphite structure size: Not etched 100X

Graphite Size							
8	7	6	5	4	3	2	1
90.8	9.1	0.1	0.0	0.0	0.0	0.0	0.0

Graphite Form Type					
I	II	III	IV	V	VI
0.0	0.0	0.1	0.7	8.0	91.2



Graphite structure form: Not etched 100X



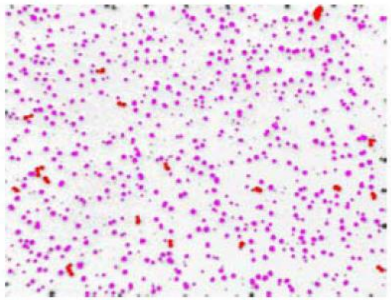
Matrix : 2% Nital etched 100X

Sample related Measurement Data			
Date of Evaluation:	06.05.2008 11:43	Phase fraction Graphite [%]:	6.55
Standard for Evaluation:	EN ISO 945	Number of Particles (absolute):	5254
Measurement fields unetched:	8	Particle Density [1/mm²]:	1660
Measurement fields etched:	0	Size Class:	6
Sample area unetched [mm²]:	3.16	Nodularity Index [%]:	63.8
Sample area etched [mm²]:		Ferrite/Pearlite Ratio [%]:	

Inclusion size/count displayed against determination errors

Structure analysis according to EN ISO 945

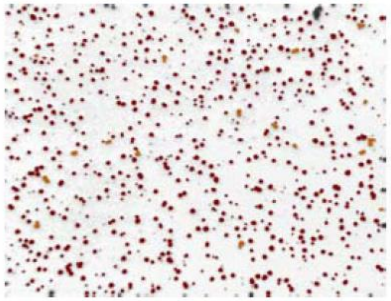
Graphite structure: Not etched 100X



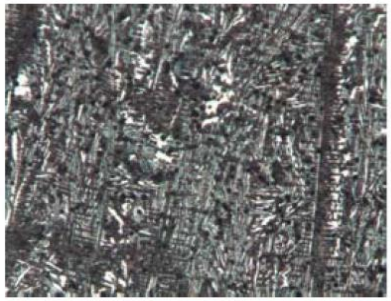
Graphite structure size: Not etched 100X

Graphite Size							
8	7	6	5	4	3	2	1
90.8	9.1	0.1	0.0	0.0	0.0	0.0	0.0

Graphite Form Type					
I	II	III	IV	V	VI
0.0	0.0	0.1	0.7	8.0	91.2



Graphite structure form: Not etched 100X



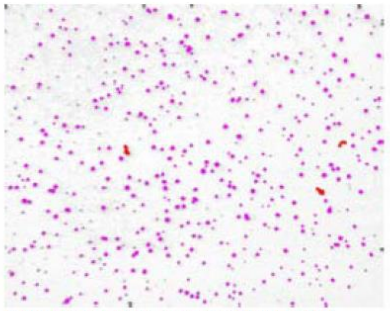
Matrix : 2% Nital etched 100X

Sample related Measurement Data			
Date of Evaluation:	06.05.2008 11:43	Phase fraction Graphite [%]:	6.55
Standard for Evaluation:	EN ISO 945	Number of Particles (absolute):	5254
Measurement fields unetched:	8	Particle Density [1/mm²]:	1660
Measurement fields etched:	0	Size Class:	6
Sample area unetched [mm²]:	3.16	Nodularity Index [%]:	63.8
Sample area etched [mm²]:		Ferrite/Pearlite Ratio [%]:	

3.07% C found, true value 3.63% C

Structure analysis according to EN ISO 945

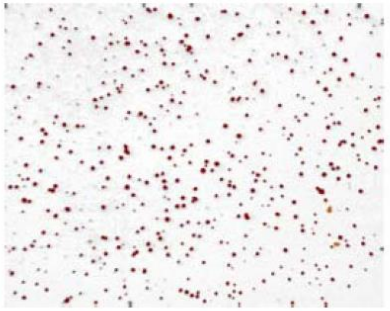
Graphite structure: Not etched 100X



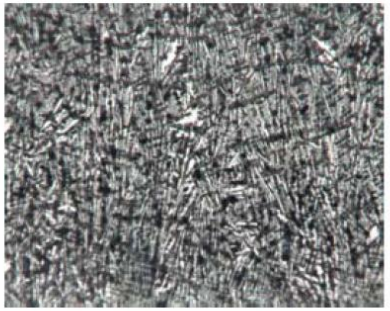
Graphite structure size: Not etched 100X

Graphite Size							
8	7	6	5	4	3	2	1
95.5	4.5	0.0	0.0	0.0	0.0	0.0	0.0

Graphite Form Type					
I	II	III	IV	V	VI
0.0	0.0	0.0	0.1	3.8	96.1



Graphite structure form: Not etched 100x

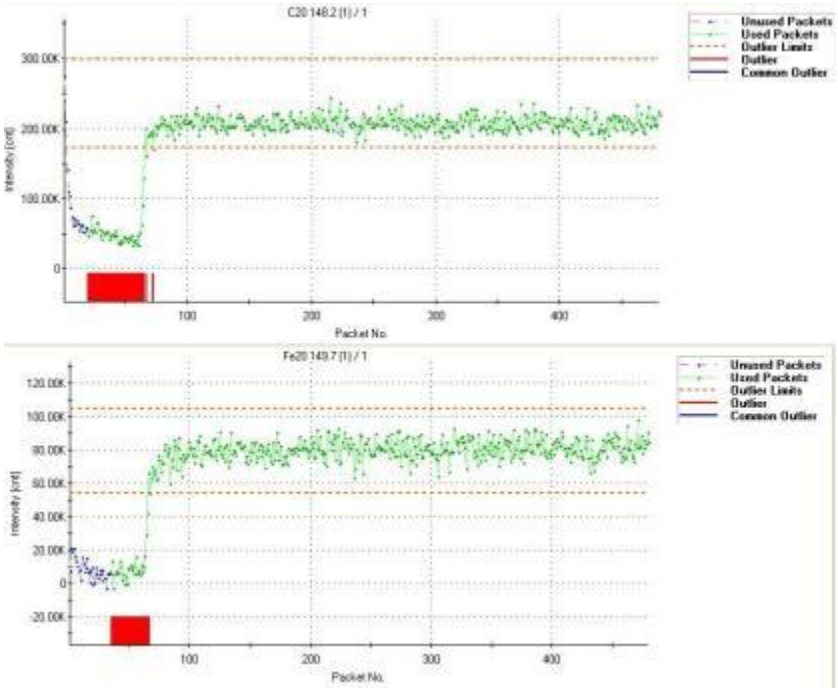


Matrix : 2% Nital etched 100X

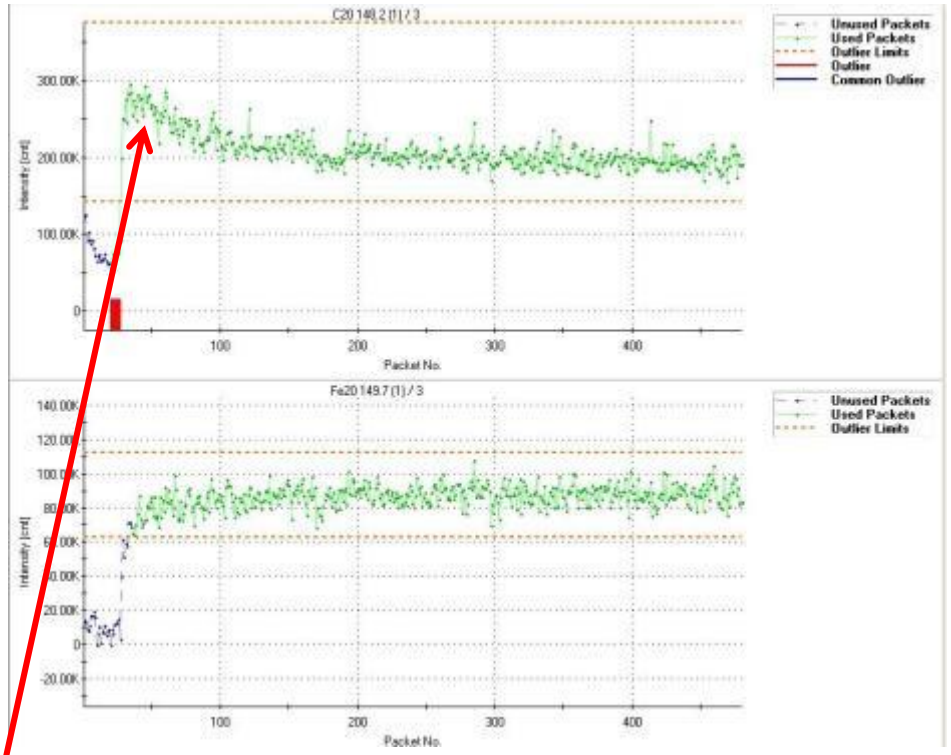
Sample related Measurement Data			
Date of Evaluation:	06.05.2008 11:46	Phase fraction Graphite [%]:	3.28
Standard for Evaluation:	EN ISO 945	Number of Particles (absolute):	3563
Measurement fields unetched:	8	Particle Density [1/mm²]:	1126
Measurement fields etched:	0	Size Class:	7
Sample area unetched [mm²]:	3.16	Nodularity Index [%]:	62.5
Sample area etched [mm²]:		Ferrite/Pearlite Ratio [%]:	

3.46% C found, true value 3.65% C

Intensities of subsequent sparks recorded during ***preburn***



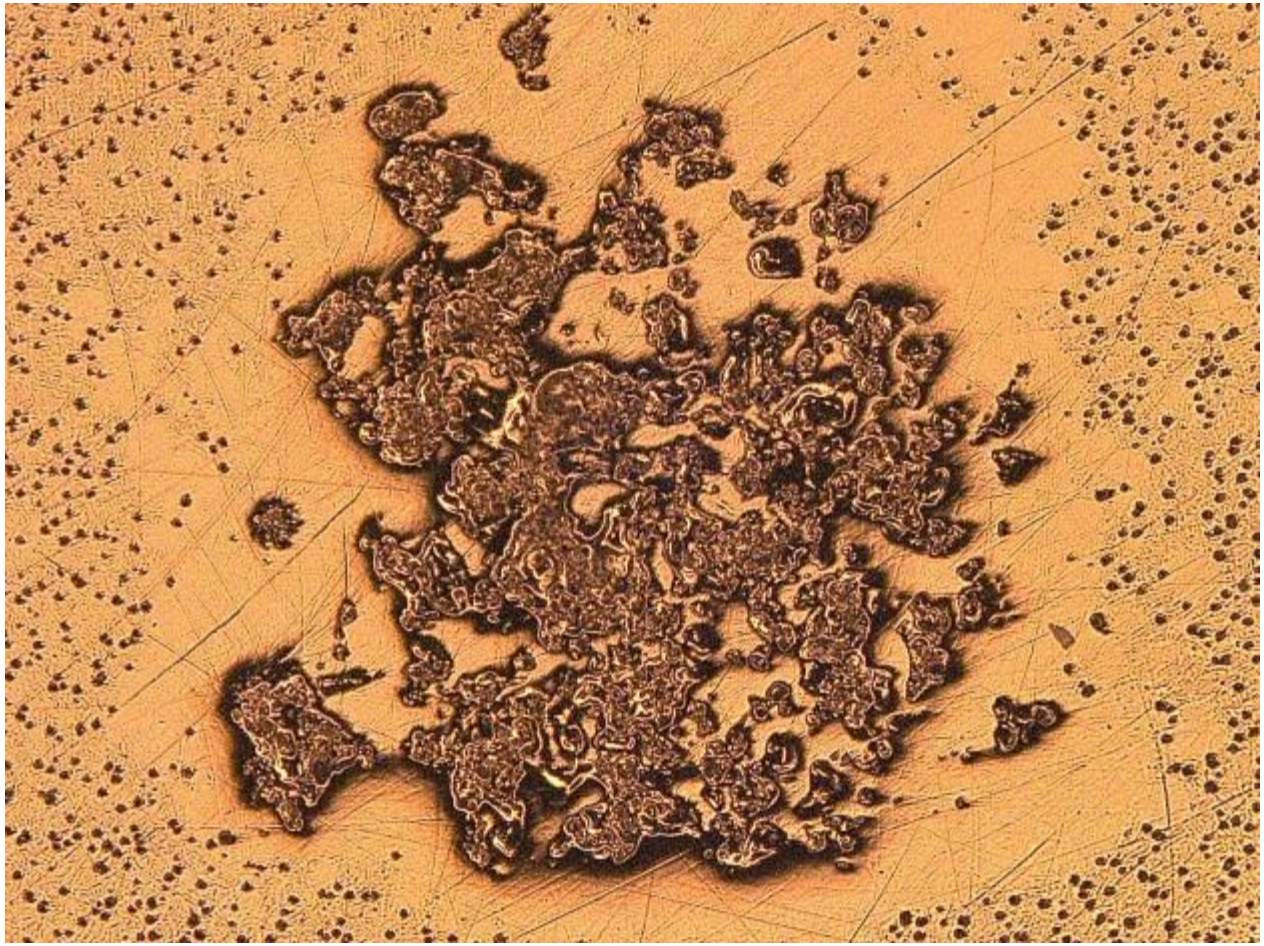
Completely white sample (*SUS5/45)*



Sample containing approx. 0.5% free graphite

This is an example of the impact of free graphite

Micrograph of a burn spot after one preburn period



The free graphite has been eliminated in the area around the preburn spot!

Conclusions:

- The spark craters are big (approx. 30µm) compared to the graphite inclusions (6-10µm)
- Statistically there is a big chance, that one spark covers a whole inclusion
- Graphite does not intrude the surrounding metal
- A spark hit graphite inclusion sublimates
- **The total carbon is depleted after preburn**

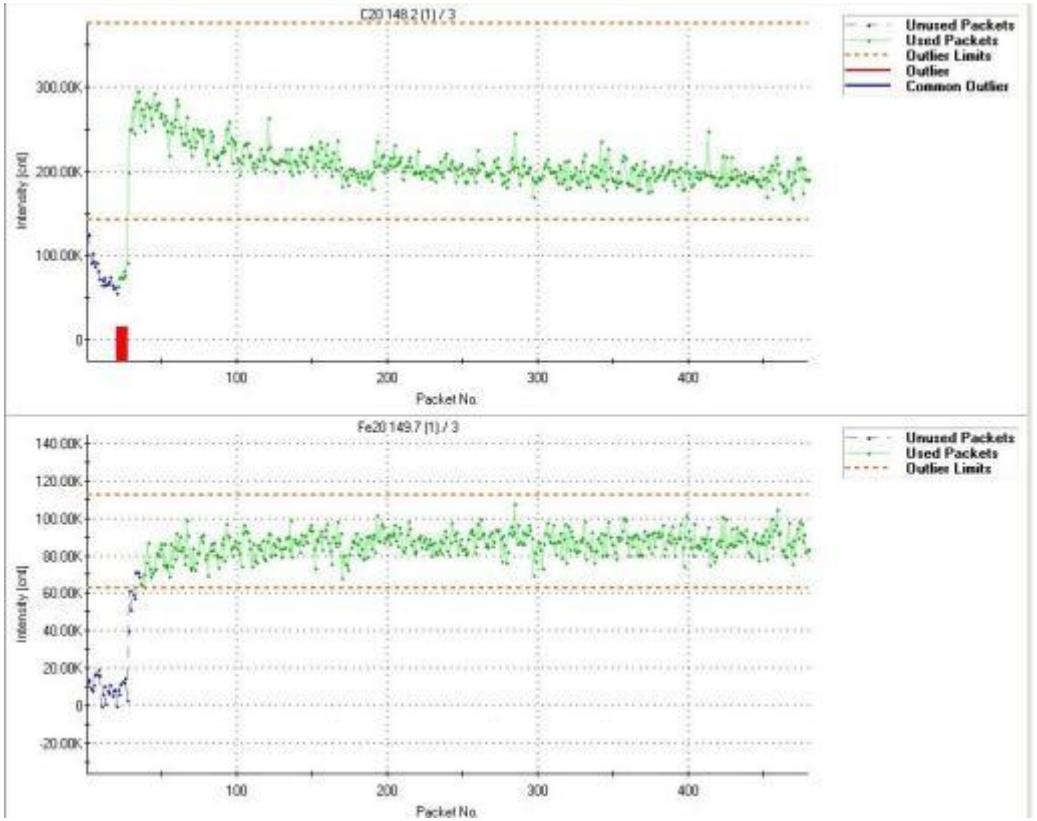
Reminder: our objectives

- Detection of bad samples
- Enabling analysis of suboptimal samples

… now the facts are clear, a straightforward solution will be elaborated

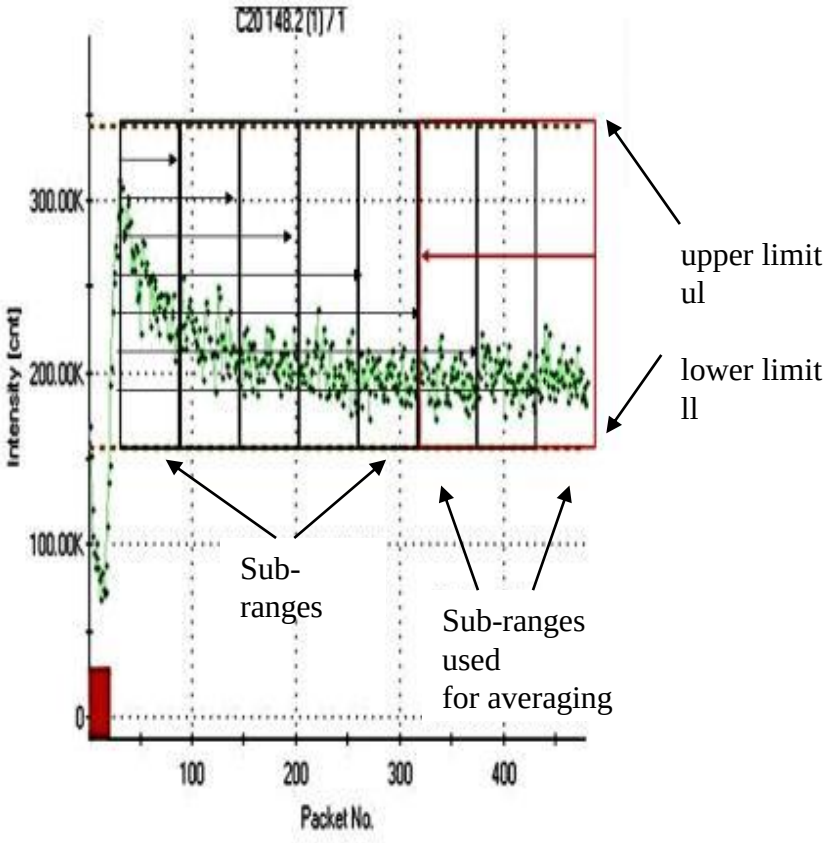
Step 1 – 4 to create a mathematical description

- Carbon and Iron are registered during pre-burn
- A small number of subsequent spark intensities (3-10) are integrated
- These integrals are called packets in our context



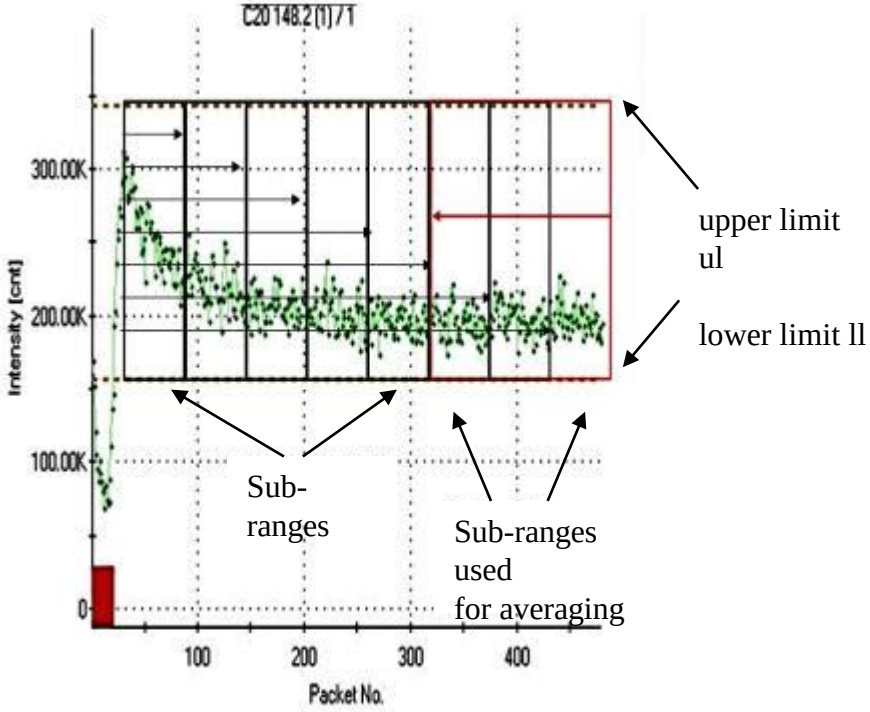
Step 2

- The last 3/8 of all packets are use to calculate average “m” and standard deviation “sd”.
- All packets before the first exceedance of “m” are ignored.
- The remaining packets are divided in a fixed number of phases “p” (p=8 in the right side example), every phase consists of “n” packets (n=55 in our example)
- For both C and Fe there is the same phase-fragmentation



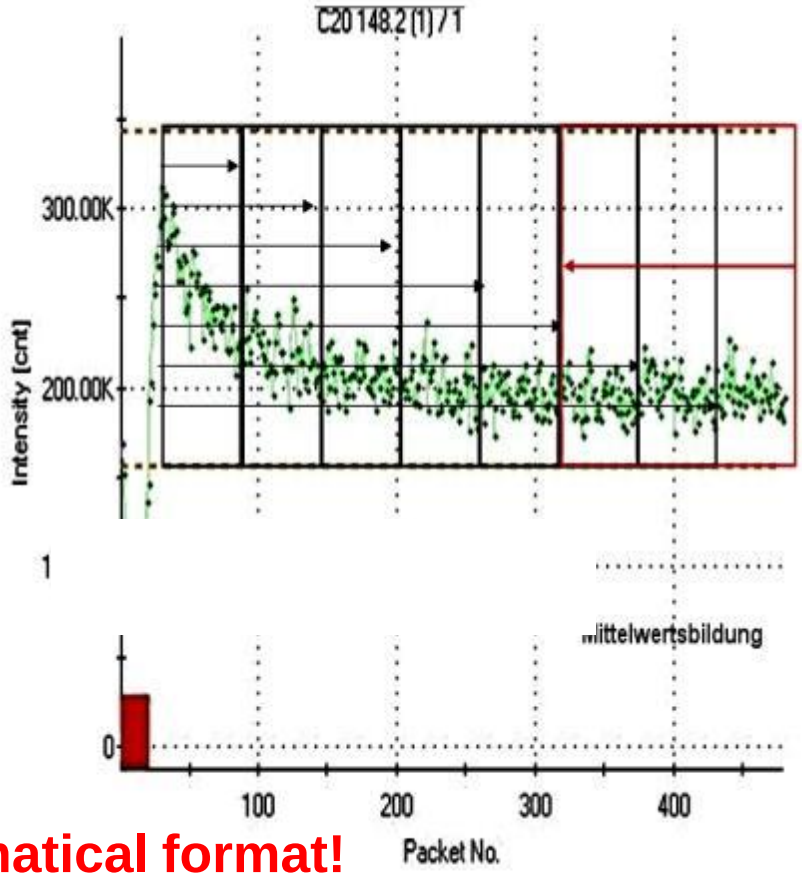
Step 3

- “sd” is used to calculate an upper limit “ul” and a lower limit “ll”
- Packets < “ll” or > “ul” are rejected
- If more than 20% of all packets are rejected, the whole burn is cancelled



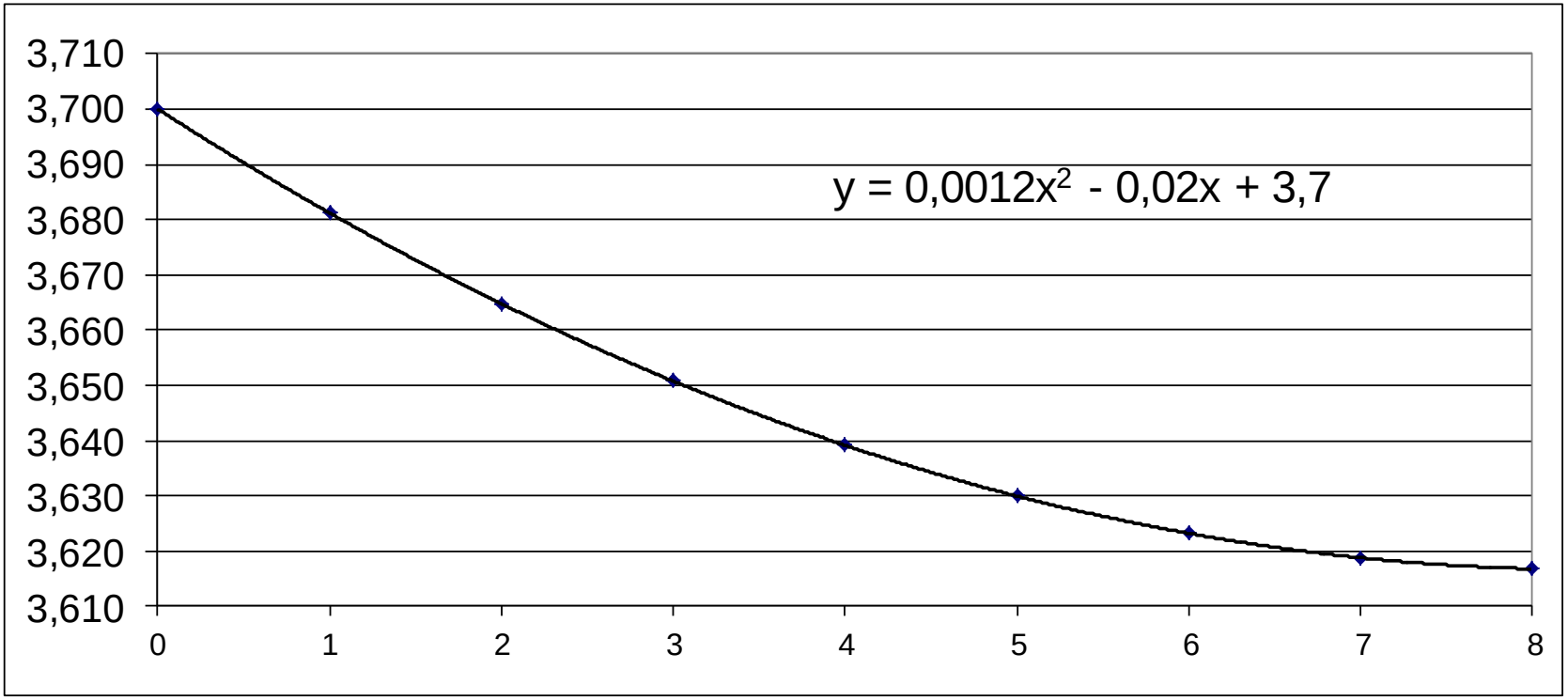
Step 4

- From phases 1, 1+2, 1+2+3, 1+2+3+4, and so on, a series of eight concentration values are calculated.
- On the series of concentration a quadratic polynomial fit is calculated
- A characteristic number k is determined from the coefficients of the polynomial: **k=-a1*a2**



These are the basics to create a mathematical format! (see next slide)

Illustration of k



$$k = - (-0.02*0.0012)$$

Results

Sample	C (conventionally measured by S-OES)	C (determined using new method of this paper)	C (combustion)
1a (slowly chilled, <= 0.2% graphite)	3.54	3.63	3.623
1b (very slowly chilled, >=0.5 % graphite, same heat as sample 1a)	3.19	3.64	3.679
Difference	**0.350**	**-0.010**	**-0.056**
8a (slowly chilled, <= 0.2% Graphit)	3.58	3.66	3.589
8b (very slowly chilled, >=0.5 % graphite, same heat as sample 8a)	3.29	3.62	3.664
Difference	**0.290**	**0.040**	**-0.075**
16a (slowly chilled, <= 0.2% graphite)	3.52	3.69	3.599
16b (grey, >=0.5 % graphite, same heat as sample 16a)	3.15	3.68	3.666
Difference	**0.370**	**0.010**	**-0.067**

Resume & Benefits

- It is possible to detect if there is any free graphite in real life samples.
- A warning occurs, as soon as a sample contains a critical level of graphite.
- C-determination errors induced by graphite are reduced (difference to combustion methods typ. <0.1%)

- => The analytical result of carbon is correct and reliable.
- If no result is possible, the operator can directly take action.

However: the method is **not** designed to analyse C in manufactured articles!

Additional information are available:

For the „White Paper“ please register, the related video is available under spectro.com!

Thank you for your time and attention