



Tin Bath Blocks & Assessment Studies

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MATERIAL ANALYSIS & CHARACTERIZATION MANAGEMENT

TC 11 – 19th of September 2022

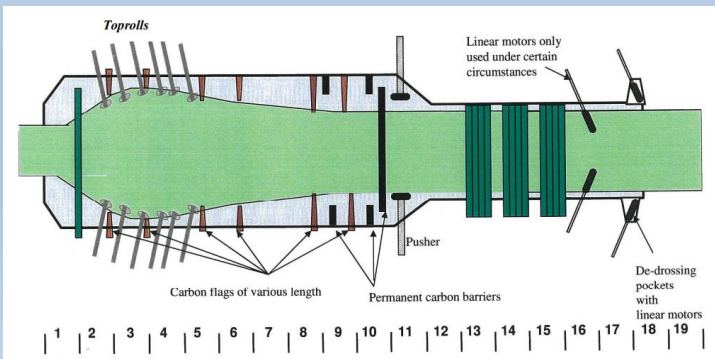
OUTLINE

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2. Expectations from Bath Blocks
3. Typical Problems Experienced by Now
4. Relationship between Material Properties and Typical Bath Block Problems
5. Examinations
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 2. Bottom Surface Bubbles
 3. Splitting (7 inches effect)
6. Results & Discussions

1. Şişecam Float Furnaces & Tin Baths



Float Furnaces in Ankara / Türkiye



Schema of Tin Baths

- 12 float lines;**
- 8 in Türkiye
 - 2 in Europe
 - 2 in Asia



Tin Bath Photos

2. Expectations from Bath Blocks

1. Chemical

- Being inert
- Without impurities
- Enough glassy phase consisting
- Resistance against alkaline attack

2. Physical

- Uniform pore size distribution
- Low gas permeability

3. Thermal

- Low thermal expansion & conductivity
- Good thermal shock resistance

4. Mechanical

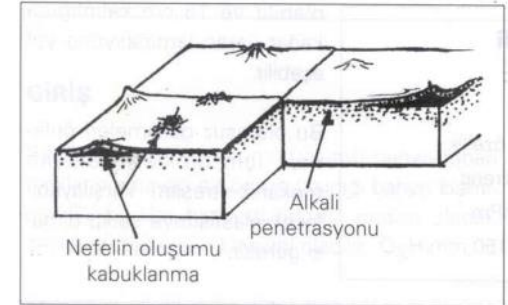
- Cold Crushing Strength
- Refractoriness Under Load

3. Typical Problems Experienced by Now

Most popular issues at float furnaces can be listed as;

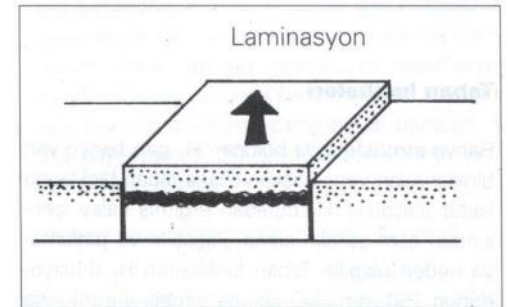
- **Flaking:**

Formation of nepheline where the alkaline penetration resistance of bath blocks are weak.



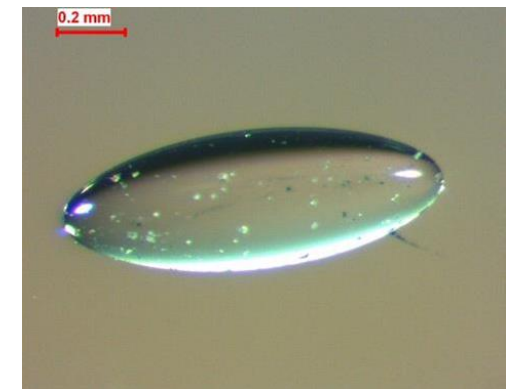
- **Lamination:**

Propagation of horizontal cracks due to internal stresses & also propagation of nepheline formation



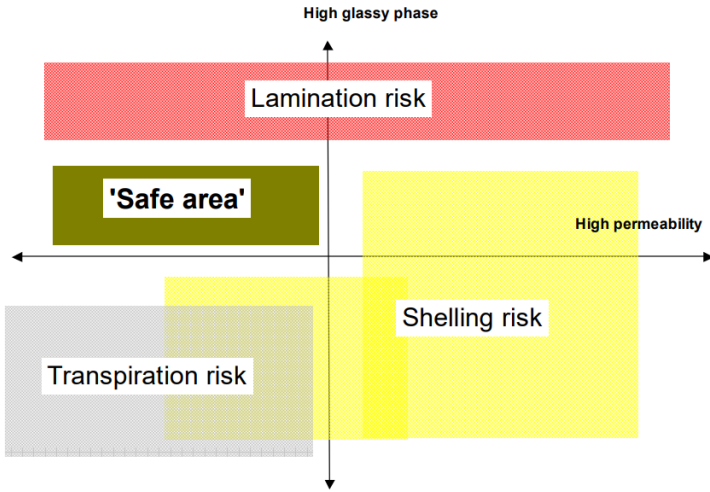
- **Bottom Side Bubbles:**

Result of high H₂ diffusion through the molten tin layer.



4. Relationship between Material Properties & Bath Block Problems

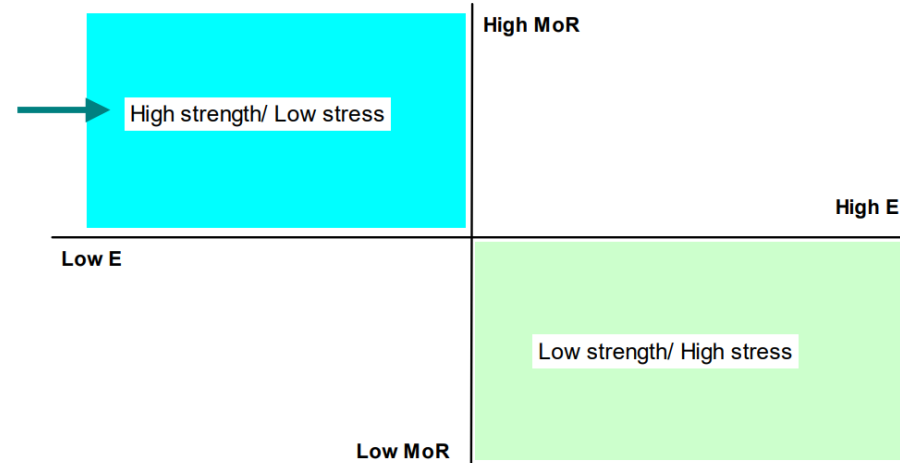
Chemical & Crystallographic Structure



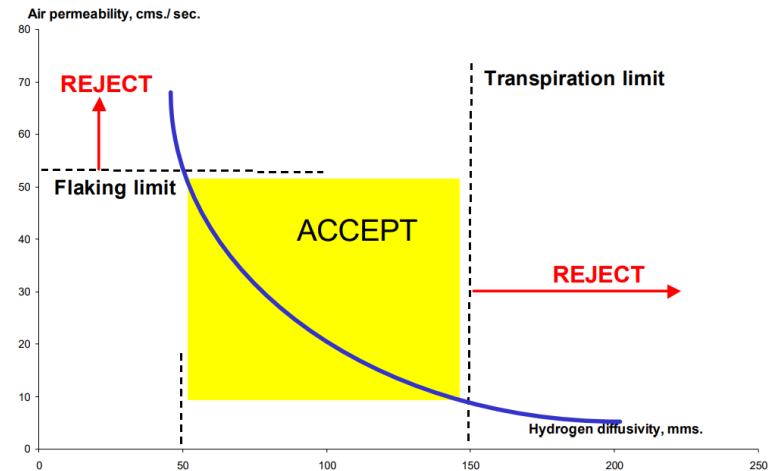
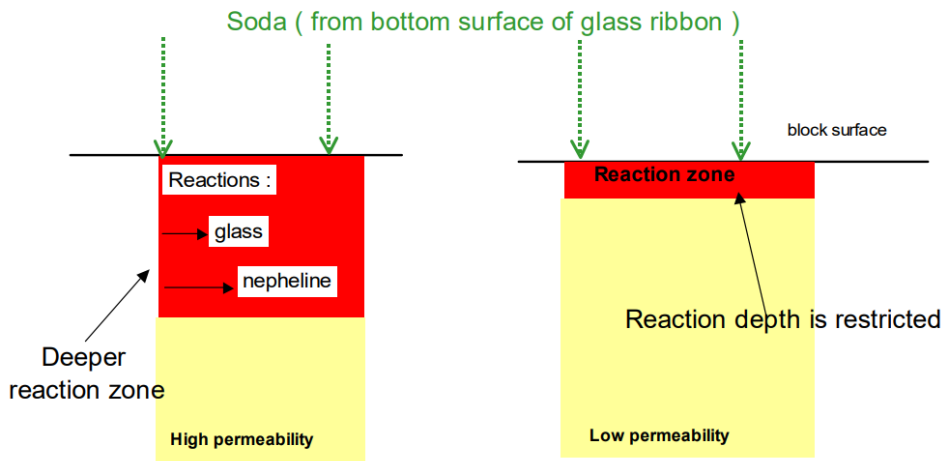
Alkaline Resistance

Cold Crushing Strength

'Lower risk' of block lamination ~ (M_{or} / E)



H2 Diffusivity



5. Examinations Depending on the Sample Types

Samples are mainly divided into 3 groups as;

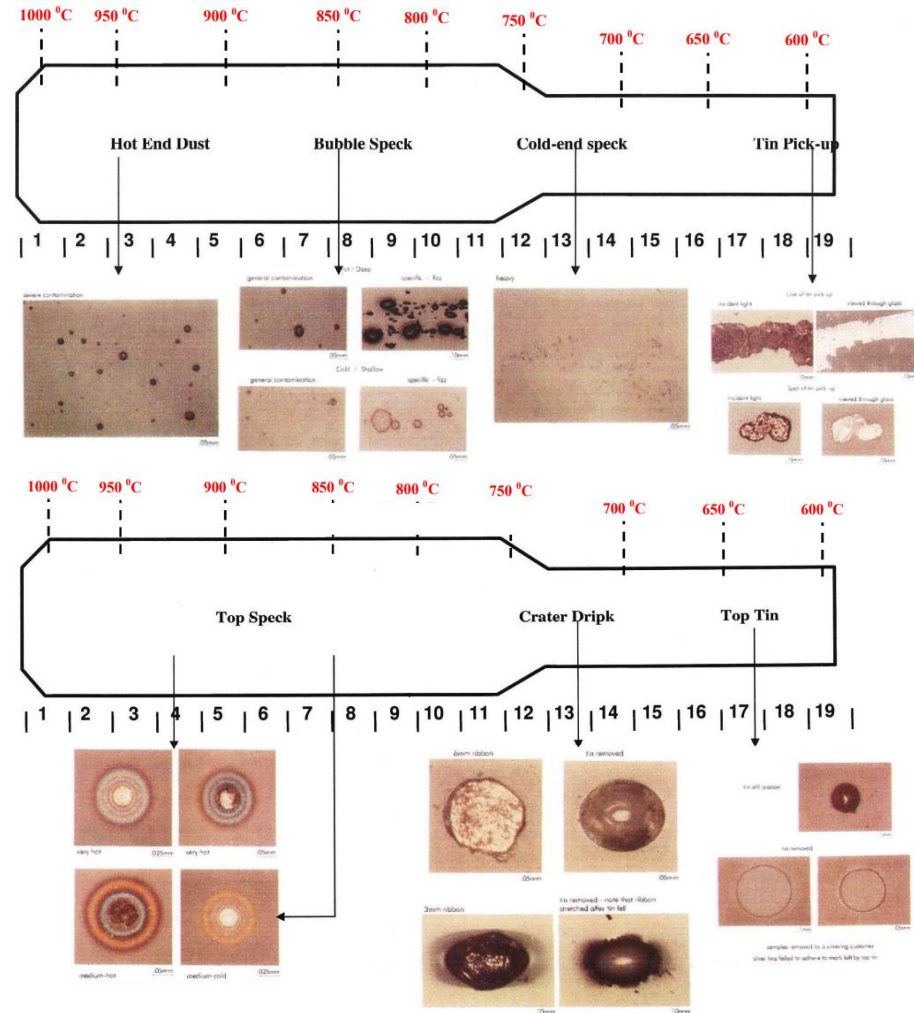
a. Postmortem samples



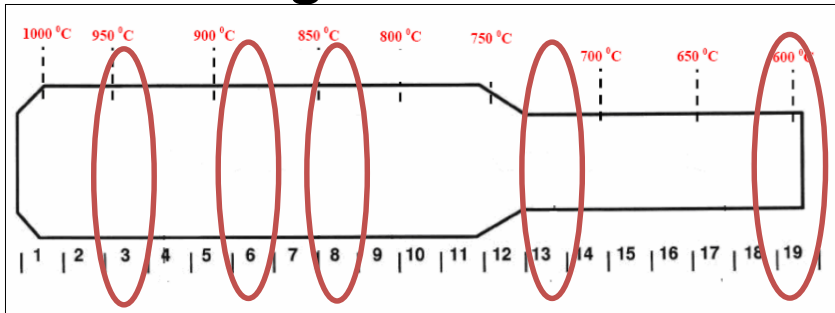
c. Non-used samples



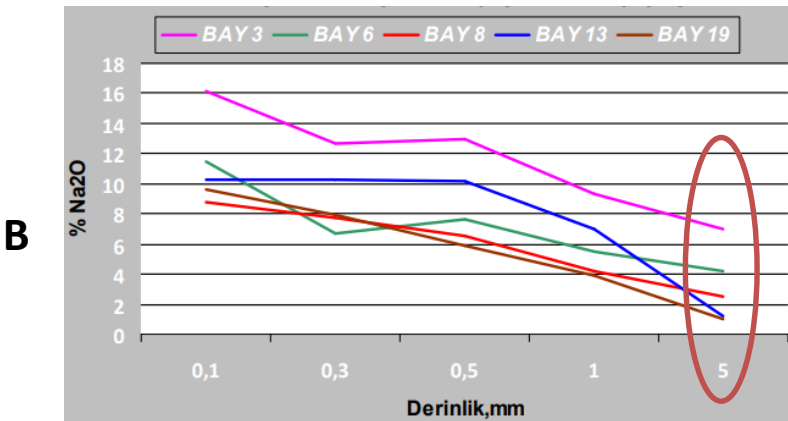
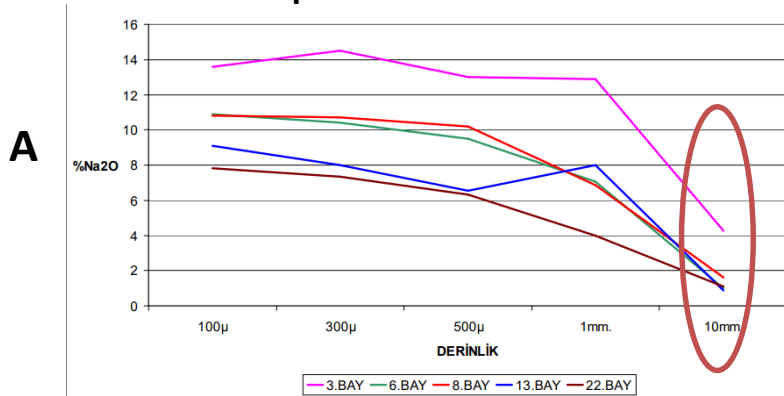
b. In-use samples (glass defects)



5.1. Flaking & Lamination



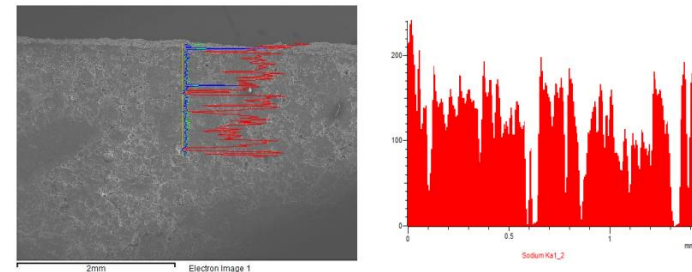
Samples taken from tin bath



Alkaline diffusion rates of A & B campaigns



Images After Campaign Life



Spectrum	Derinlik, mm	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃	SnO ₂
Spectrum 1	0.1	20.41	0.18	31.97	44.72	0.77	0.31	1.16	0.09	0.38
Spectrum 2	0.3	18.33	--	34.49	43.56	0.59	0.36	1.31	0.13	1.23
Spectrum 3	0.5	8.78	--	39.99	48.32	0.37	0.11	1.18	0.42	0.84
Spectrum 4	1	15.04	0.14	29.03	53.44	0.47	0.25	0.94	0.48	0.20
Spectrum 5	10	7.54	0.22	32.16	57.33	0.73	0.32	1.07	0.56	0.08

EDX Analyzes of Linescan Mode

5.1. Flaking Assessments (non-used Samples)

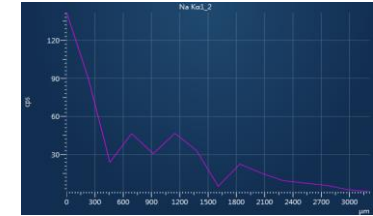


Alkaline penetration test set up

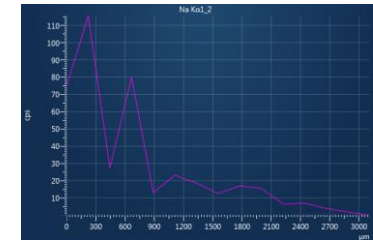


Cross-sectional view

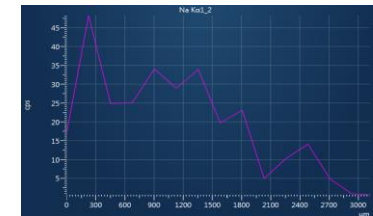
Sample 1



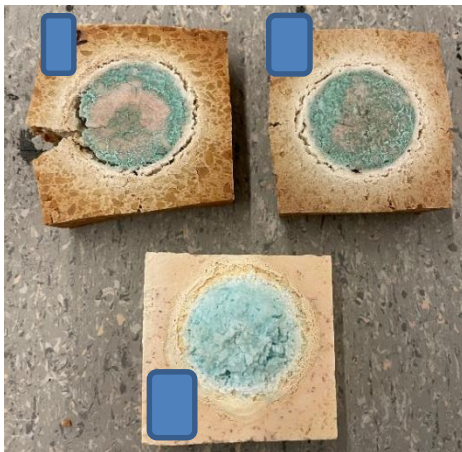
Sample 2



Sample 3



EDX line scans of each sample (%Na₂O only)



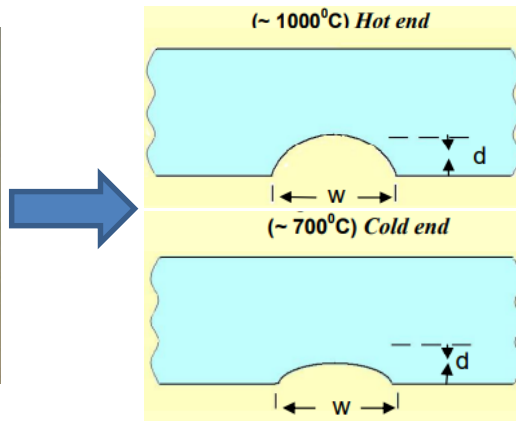
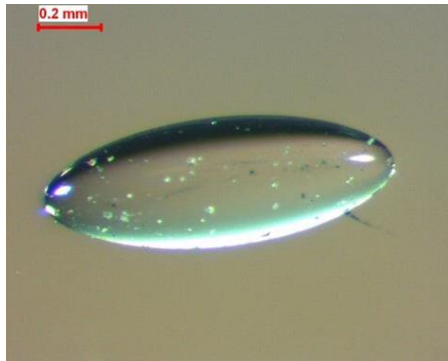
Bottom View

% Na₂O Amounts of each sample via EDX

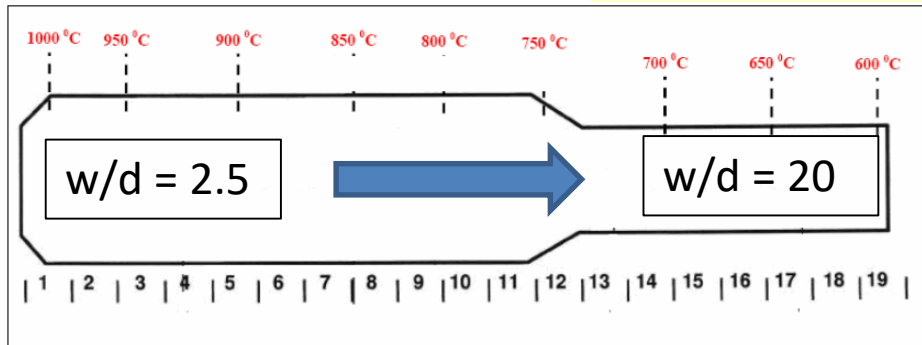
Spectrum Label	Spectrum 1	Spectrum 2	Spectrum 3	Spectrum 4	Spectrum 5	Spectrum 6	Spectrum 7	Spectrum 8	Spectrum 9	Spectrum 10	Spectrum 11	Spectrum 12	Spectrum 13	Spectrum 14
Sample 1	4,45	15,59	10,9	7,57	4,79	5,47	4,43	3,98	3,5	1,25	-	-	-	-
Sample 2	1,88	0,43	0,63	1,78	1,24	1,46	1,33	3,69	1,72	0,65	1,35	0,66	-	-
Sample 3	21,07	13,04	12,69	9,14	6,46	10,12	9,4	9,6	10,35	10,64	5,46	5,27	1,92	0,41

→→→From top surface to bulk material→→→

5.2. Bottom Surface Bubbles

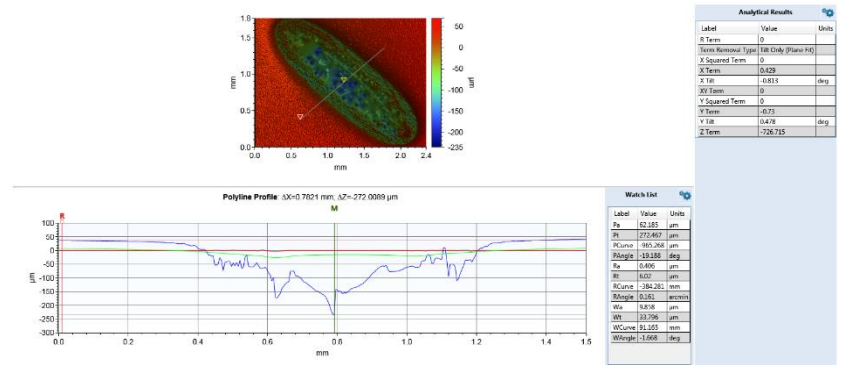
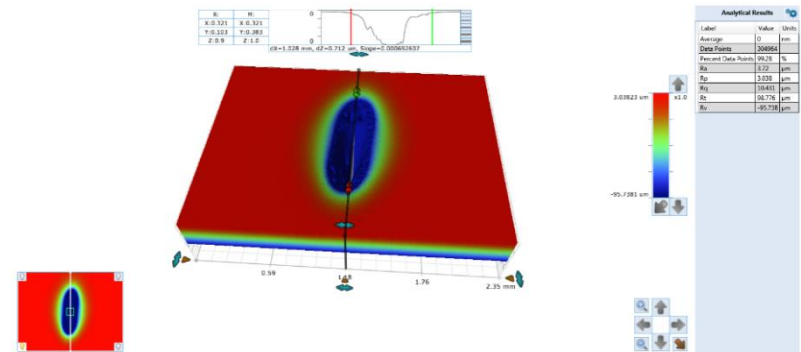


- Large open bubbles at the bottom surfaces;
- Displacement of bath atmosphere from block joints by molten tin
 - Soda migration from glass
 - Thermal transpiration (capillary effect due to the porosity of the refractories)
 - H2 coming out of molten tin.



As the w/d ratio goes from 2.5 to 20;
 The origin of the bubbles approaches to the cold end.

w/d ratio defines where the open porosities come from

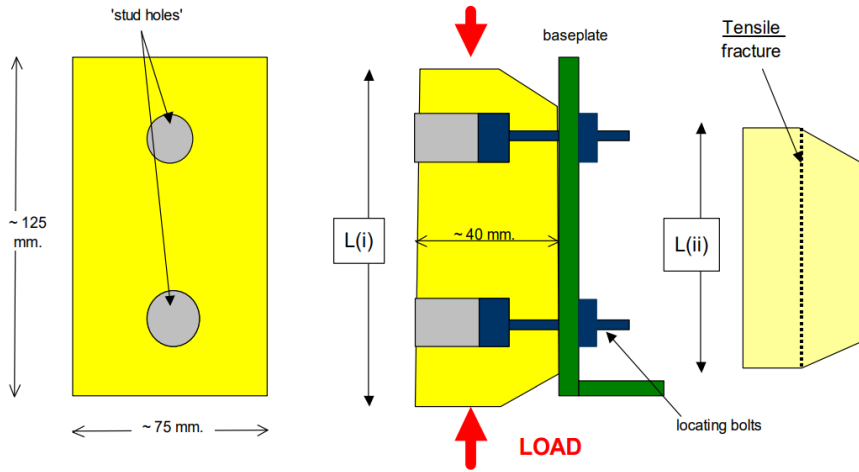


Confocal Systems for w & d measurements

5.3. Splitting (7 inch effect)

Proof Test

$$\% \text{ 'Strain to failure' } = (L(i) - L(ii)) \times 100 / L(i)$$



Cold Crushing Strength & Bending Tests



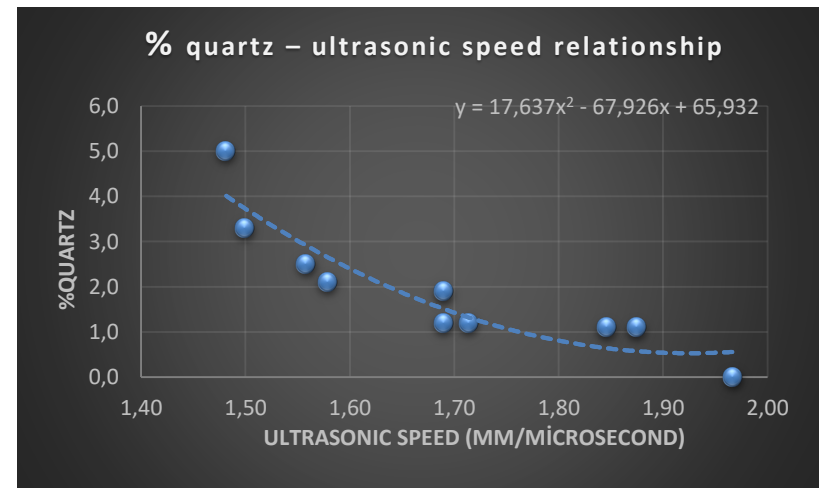
Ultrasonic Measurements



???



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6.1. Results

Main analytical techniques used in Şişecam are;

- Microscopic Techniques (SEM-EDX, Optical Microscopy)
- X-Ray Techniques (XRF, XRD)
- Confocal Systems (WLI)

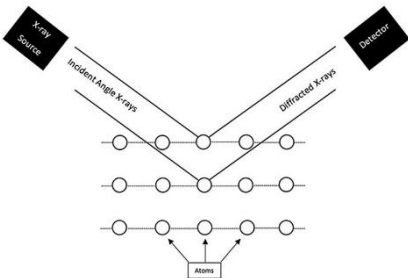
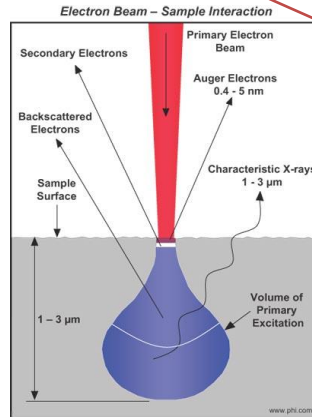
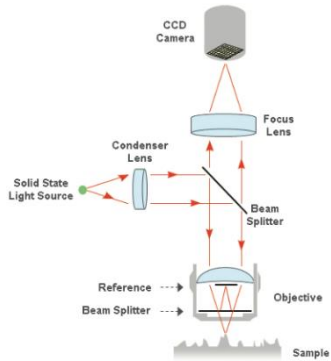
• For microstructure imaging and point EDX analyzes of refractory materials.

• For chemical & crystallographic composition of refractory materials

• For collecting data from open bubbles (w/d ratio)

• Crystallographic composition of refractory materials

To identify the defects and microstructure of the related refractories.



Main tests to be applied for tin bath blocks are;

- Alkaline penetration test
- Ultrasonic measurement
- Hydrogen Diffusivity test
- Proof test
- Cold Crushing test

To make performance estimation & comparison of non-used bath blocks.

6.2. Discussions

- Sodium penetration investigations (both analyzes from postmortem samples & tests on non-used samples) are our only way to compare and assessment mechanism. Any other way to make stronger comments on the blocks?
- Is there any non-destructive method for controlling inner impurities/cracks/voids of the bath block?
- How the effect of refractory materials on bottom surface bubbles can be estimated?
- Any applications on bath block surfaces (paving, coating etc.) can act as protecting agent of Hydrogen diffusion?