Session: Forensic Investigations of Concrete Failures Using Advanced Techniques,
Part 2 of 2

Petrographic Techniques to Identify and Determine Relative Abundance of Iron Sulfide Minerals in Concrete Aggregate

Presented by: April Snyder

OUTLINE

- Background
- Sample Preparation
- Reflected Light Microscopy
- Scanning Electron Microscopy with X-ray Mapping
-

BACKGROUND

Quebec pledges $17M more for pyrrhotite-damaged homes

Government also lowers amount of problematic mineral in foundation concrete required to access funds

By Stephen Smith, CBC News Posted: Jan 06, 2017 2:18 PM ET | Last Updated: Jan 06, 2017 7:16 PM ET

This house is one of hundreds that have undergone repairs to fix damage caused by pyrrhotite.

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Quebec is allocating $17 million more for homeowners in the Mauricie an Central Quebec regions affected by pyrrhotite.

The Liberal government announced the new funds Friday at a news conference in Trois-Rivières, 140 kilometres northeast of Montreal.

Pyrrhotite is a mineral that expands when exposed to humidity and oxygen. Its presence in aggregate can lead to cracks in concrete structures.

That problem has wreaked havoc on the foundations of thousands of homes and commercial buildings in the Mauricie and Central Quebec regions built between 1998 and 2008 with aggregate from a local quarry.

- Homeowners demand $25M from Ottawa for structural damag

Lower threshold

In addition to the new funds, the government also announced Friday that was lowering the volume of pyrrhotite in concrete that's required to acces the funds from 0.3 to 0.23 per cent.

The lower threshold and extra funds will help an estimated 400 homeowners and brings Quebec's total assistance for homeowners coping with pyrrhotite damage to $52 million.

Homeowner Marc Dubord welcomed the decision to lower the pyrrhotite threshold. The concrete in his foundation has a volume of .25 per cent

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- What's in the federal budget for Quebec, and what's missing
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Ottawa to spend $30M helping Quebec homeowners with pyrrhotite problems

Prime Minister Justin Trudeau looks at the foundation of a house, in Trois-Rivieres, Que., on Wednesda CANADIAN PRESS/Paul Chiasson)

15 15

The Canadian Press
Published Wednesday, April 6, 2016 4:10PM EDT
Last Updated Wednesday, April 6, 2016 5:46PM EDT

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ers in 23 towns have filed complaints with the state Department of Consumer Protection alleging their re failing.

Cracks in Cloutier's basement walls caused by pyrrhotite (Source: Keith Cloutier)

Fool's Foundation: The Little-Known Crisis That's Costing Homeowners Thousands—and Why Massachusetts Is Finally Paying Attention

BY ALLAIRE CONTE

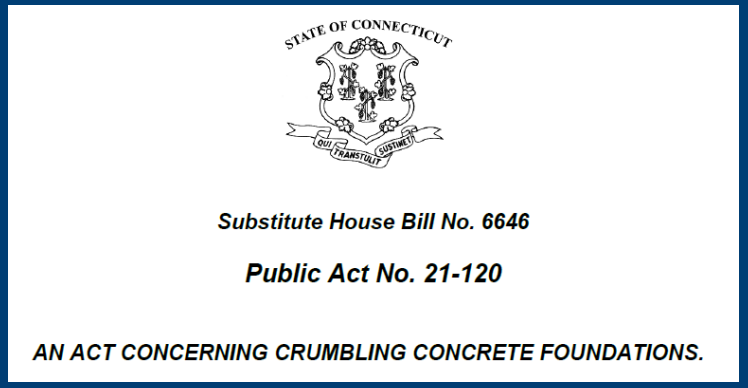
JULY 10, 2025

aci® American Concrete Institute

BACKGROUND

Legislation in Connecticut and Massachusetts to regulate iron sulfides in concrete aggregates.

- Reject if total sulfur is 1.0% or more
- Accept if total sulfur is less than 0.1%
- if 0.1% to 1.0%
  - Determine the presence and relative abundance of pyrrhotite
    - Magnetic susceptibility
    - X-ray Diffraction (XRD)
    - Petrographic Analysis, ASTM C295


STATE OF CONNECTICUT

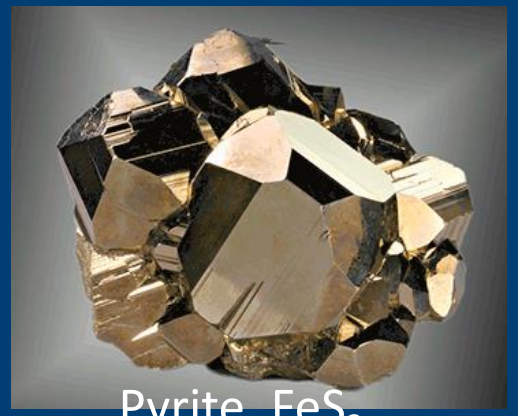
Substitute House Bill No. 6646

Public Act No. 21-120

AN ACT CONCERNING CRUMBLING CONCRETE FOUNDATIONS.

BACKGROUND Iron Sulfides

- Common accessory mineral in many forms.
  Pyrite, marcasite, pyrrhotite, chalcopyrite, pentlandite
- Found in all rock types.
- Pyrite is most common.
- Pyrrhotite is most problematic in concrete



Pyrite, FeS_2

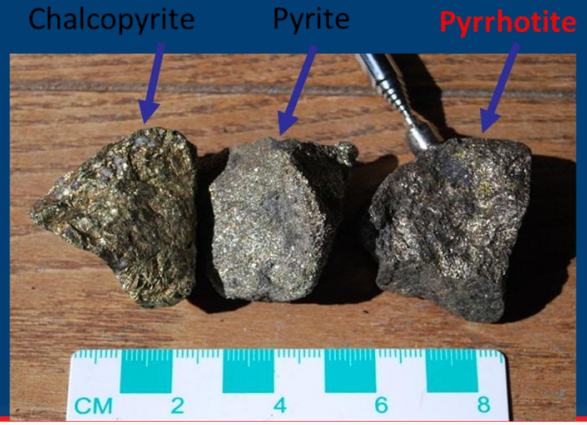
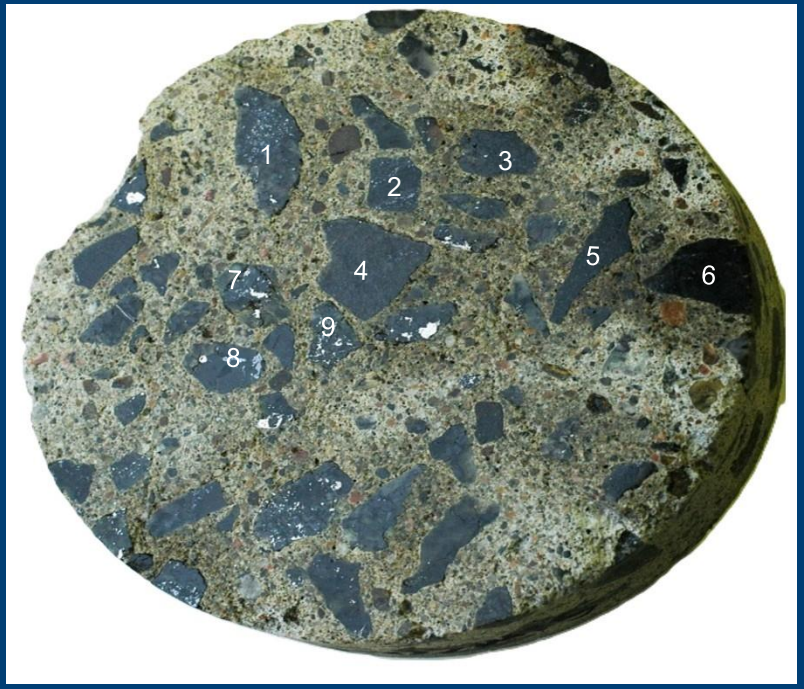


Image source: C. Rogers

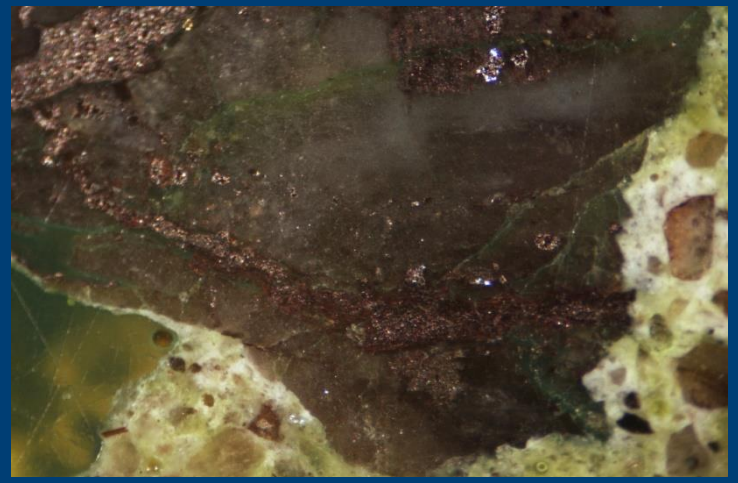
Pyrrhotite, $Fe_{(1-x)}S$, $x \leq 0.125$

Pyrrhotite in Aggregates

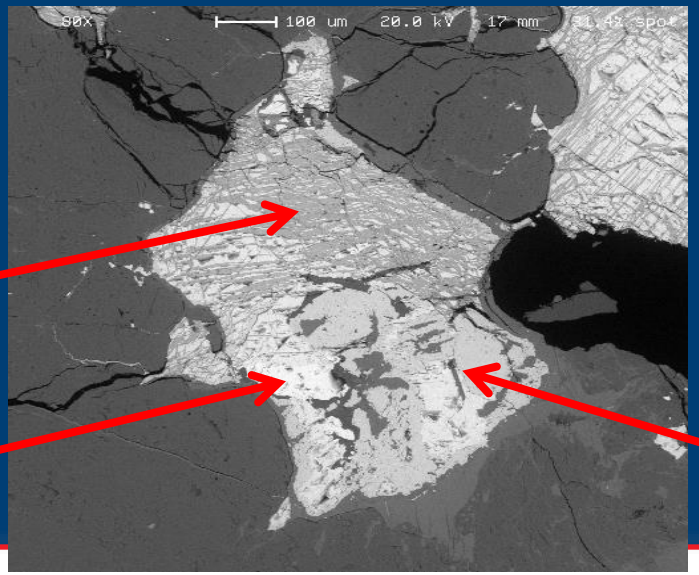
Typically appears as minor mineral inclusions



100 mm diameter



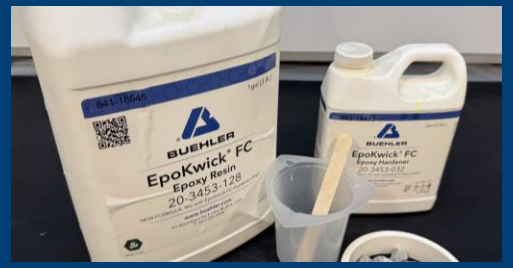
Aggregates often contain multiple iron sulfide mineral phases

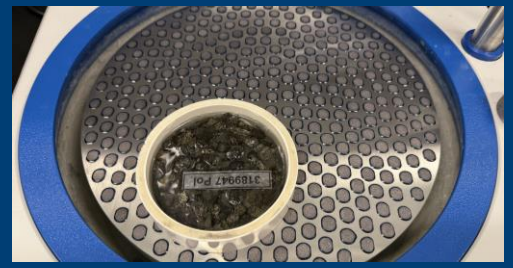


pyrrhotite

chalcopyrite

pyrite





The importance of

SAMPLE PREPARATON

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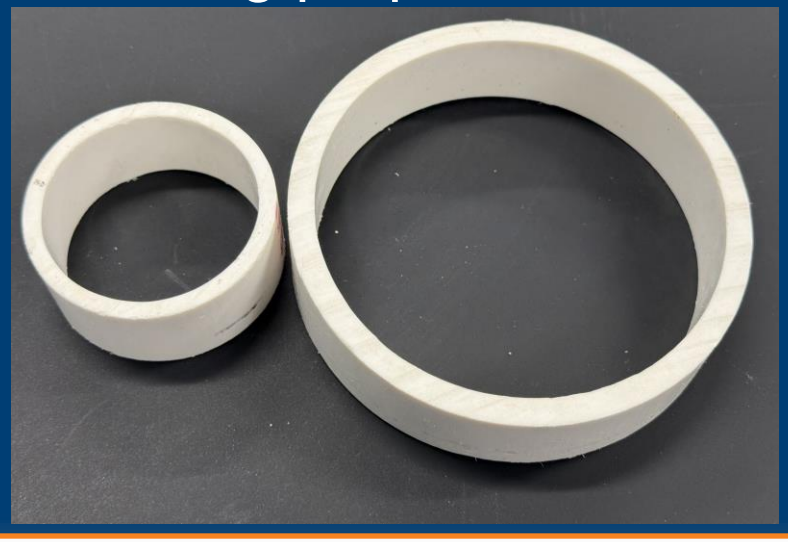
Sample Preparation

Mounting

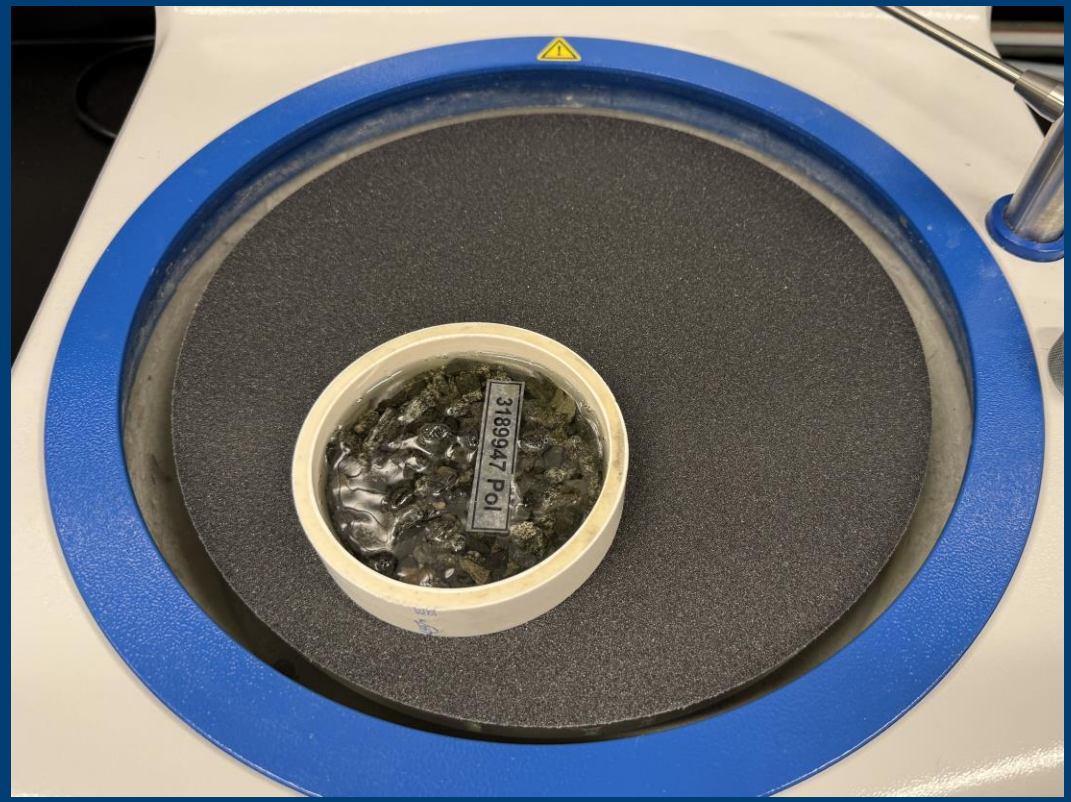
- Aggregates are embedded in a 1” PVC mold
- Provides a stable platform for grinding and polishing
- Ensures fine sulfide grains and delicate textures remain intact during preparation



Sample Preparation

Grinding

- Material is ground progressively to expose sufficient surface area
- Controlled removal prevents loss of soft mineral phases
- Ensures representative exposure of iron sulfide-bearing regions



Sample Preparation

Polishing

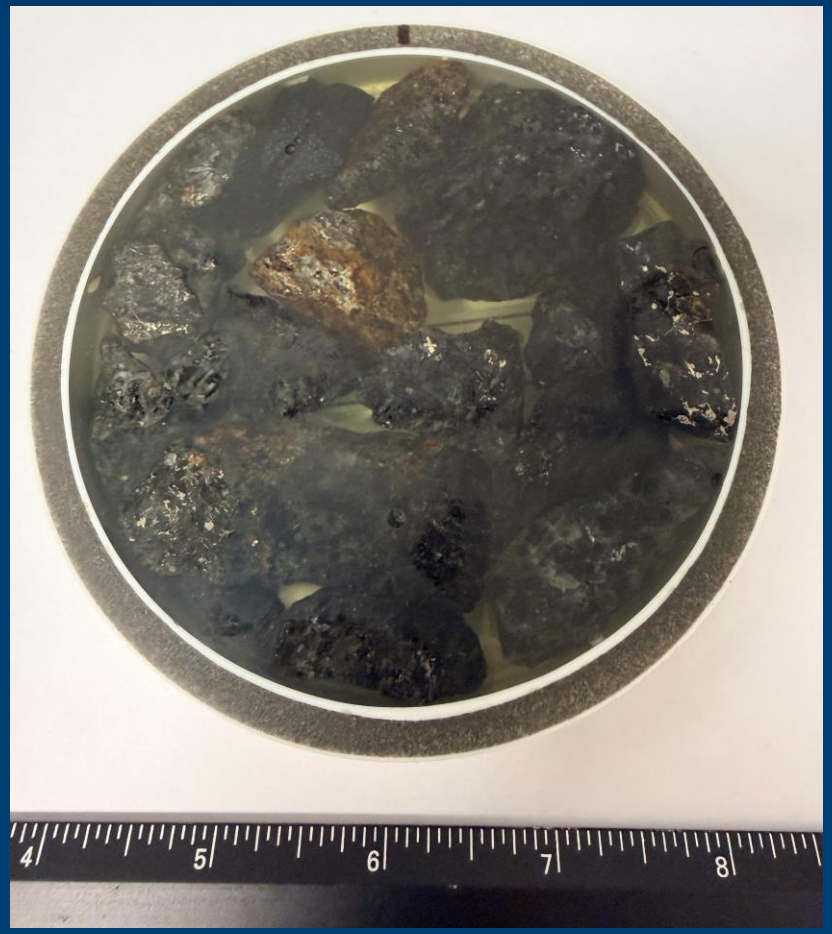
- Final polish to 0.25 µm produces a smooth surface with minimal relief
- Essential for scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS)
- Eliminates surface imperfections that can obscure fine textural or compositional details

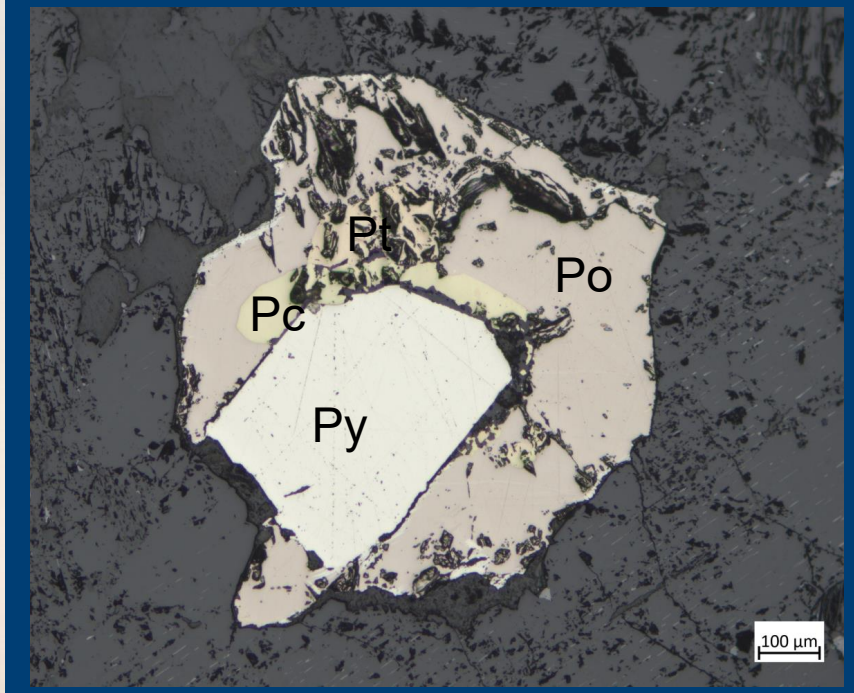
Why Quality Preparation Matters

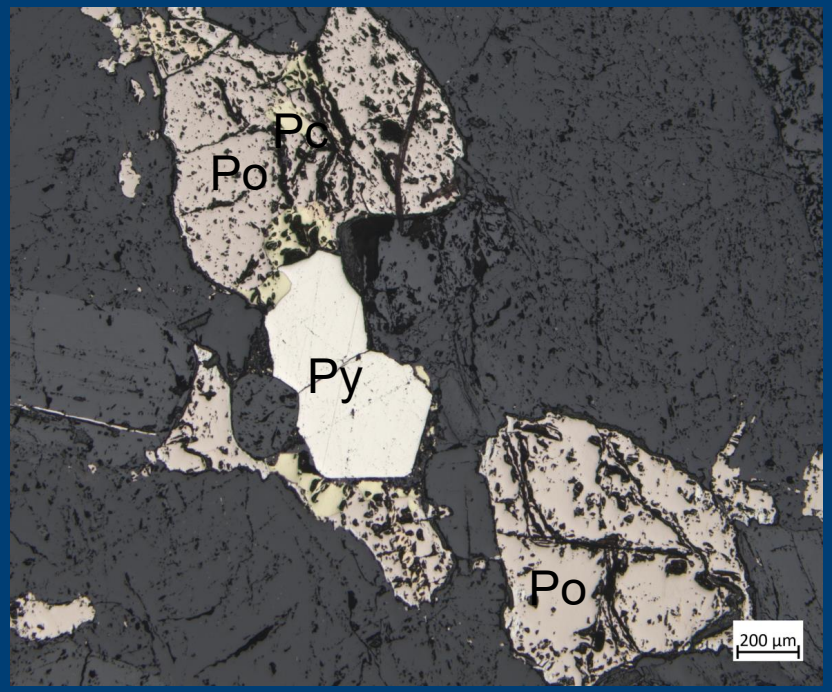
- High-quality sample preparation is critical for reliable detection and interpretation of iron sulfide minerals.
  - » Prevents misidentification due to surface artifacts or poor polish
  - » Ensures accurate imaging, compositional mapping, and phase distinction under reflected light and SEM
  - » Reveals subtle mineral associations that can influence concrete durability and performance

A well-prepared surface is the foundation of meaningful petrographic and microanalytical results

REFLECTED LIGHT MICROSCOPY



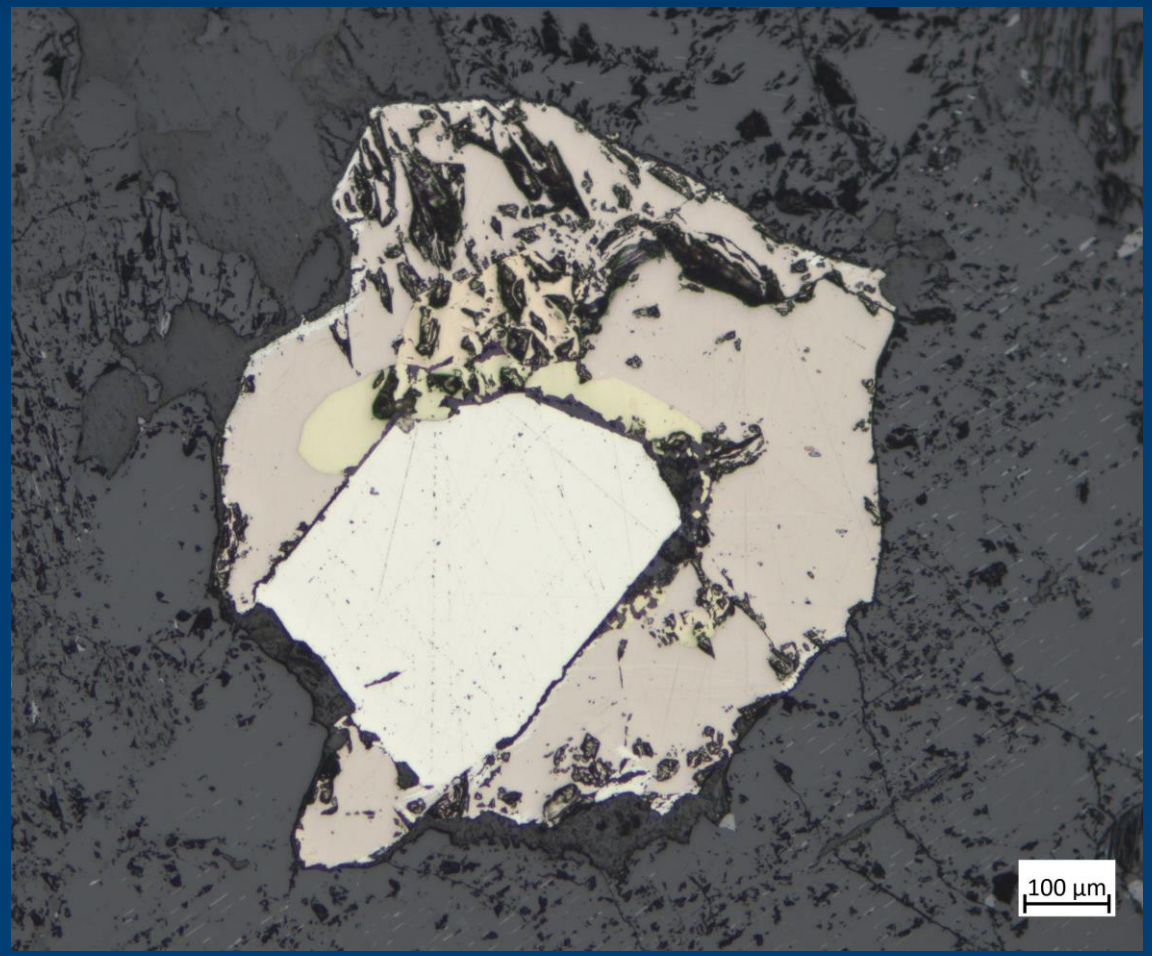


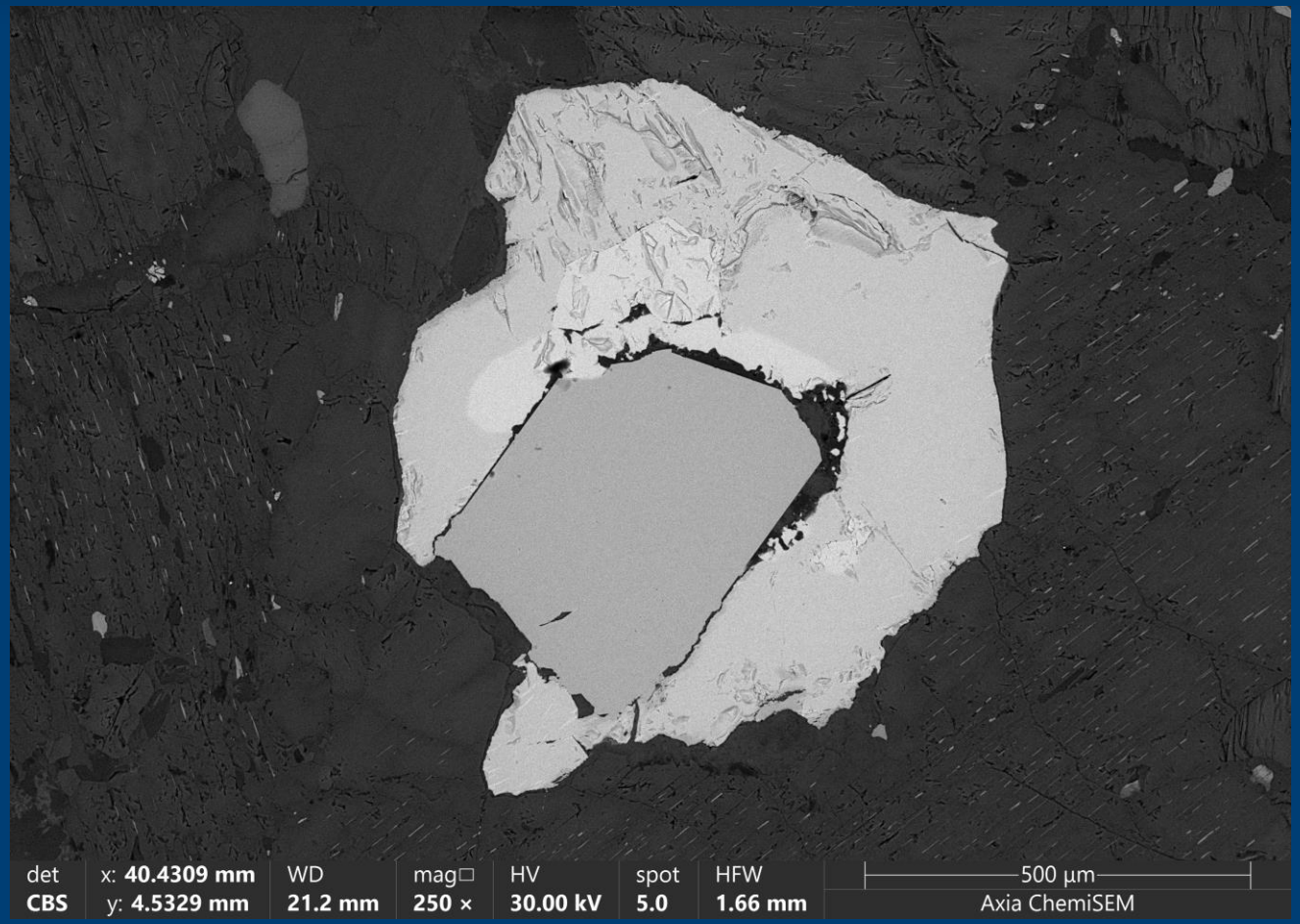


Py – pyrite
Po - pyrrhotite

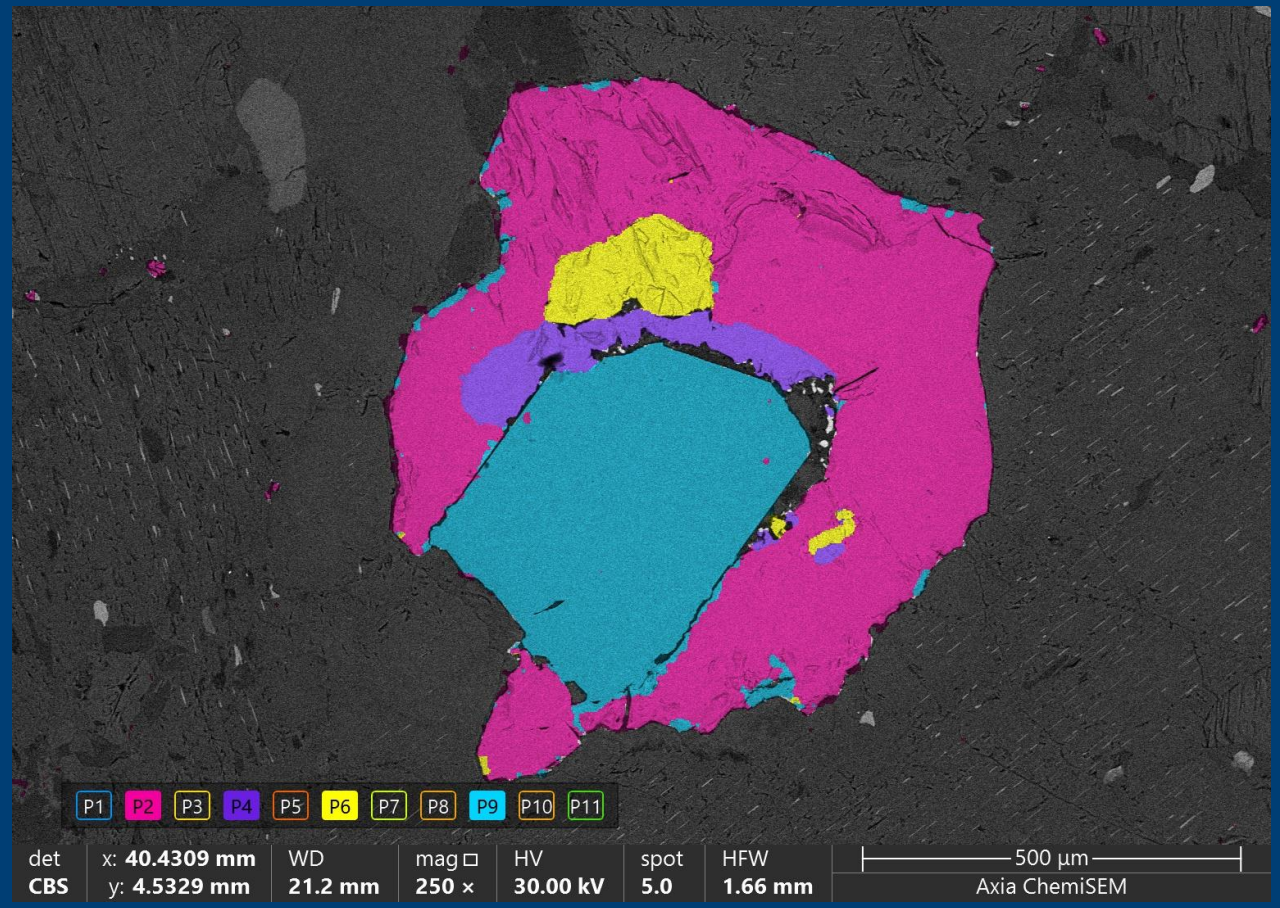
Pc – chalcopyrite
Pt - pentlandite

SCANNING ELECTRON MICROSCOPY



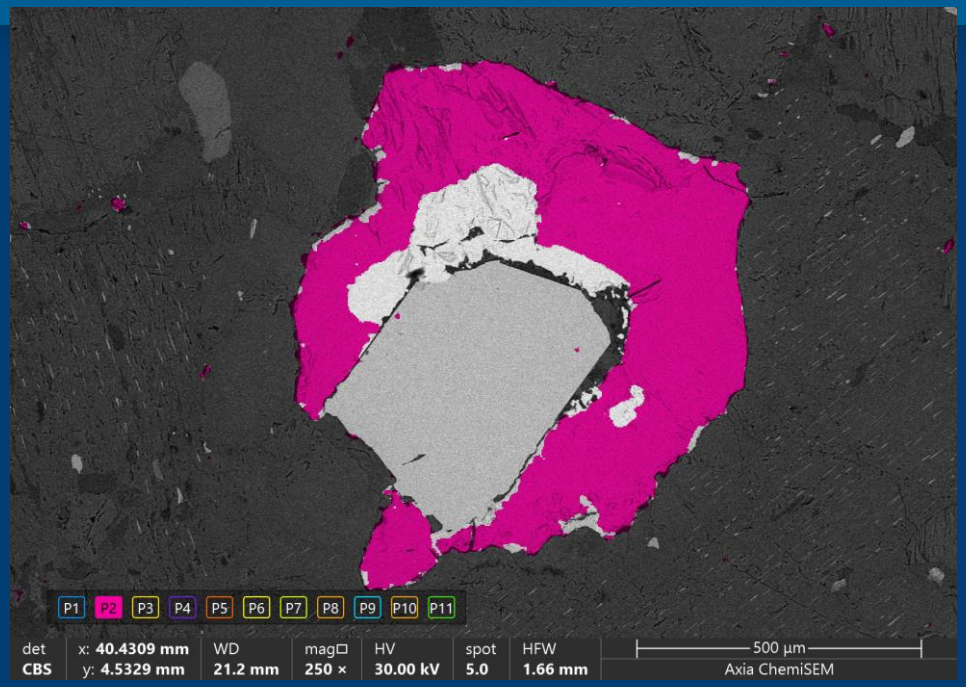


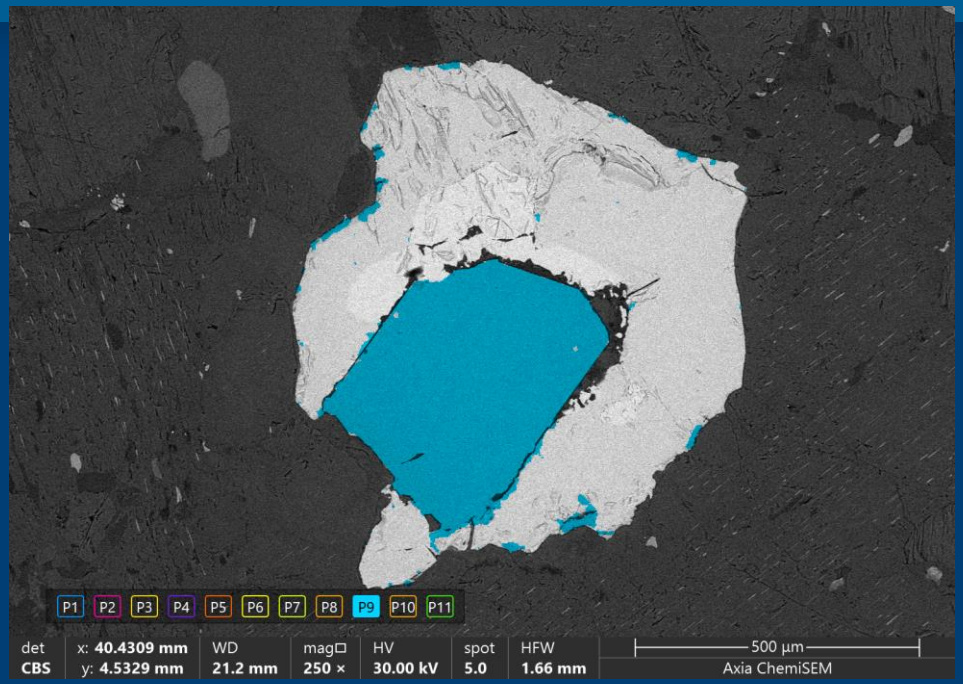
EDS X-RAY PHASE MAPPING

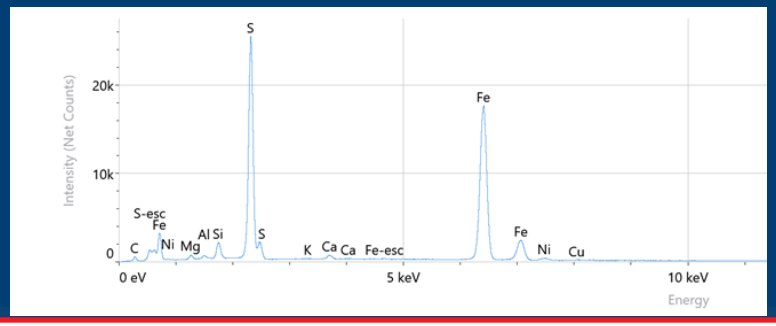


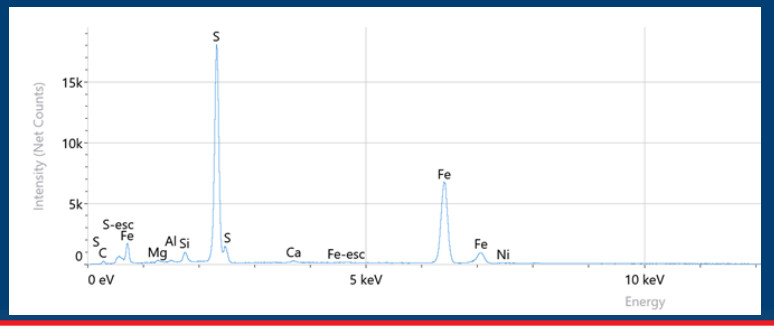
Name	Color	Area (µm²)	Area (px)	Normalized Area %
Phase 2		309574.2	66 455	70
Phase 4		28998.6	6 225	6
Phase 6		25481.5	5 470	5
Phase 9		152390.3	32 713	29

EDS X-RAY PHASE MAPPING









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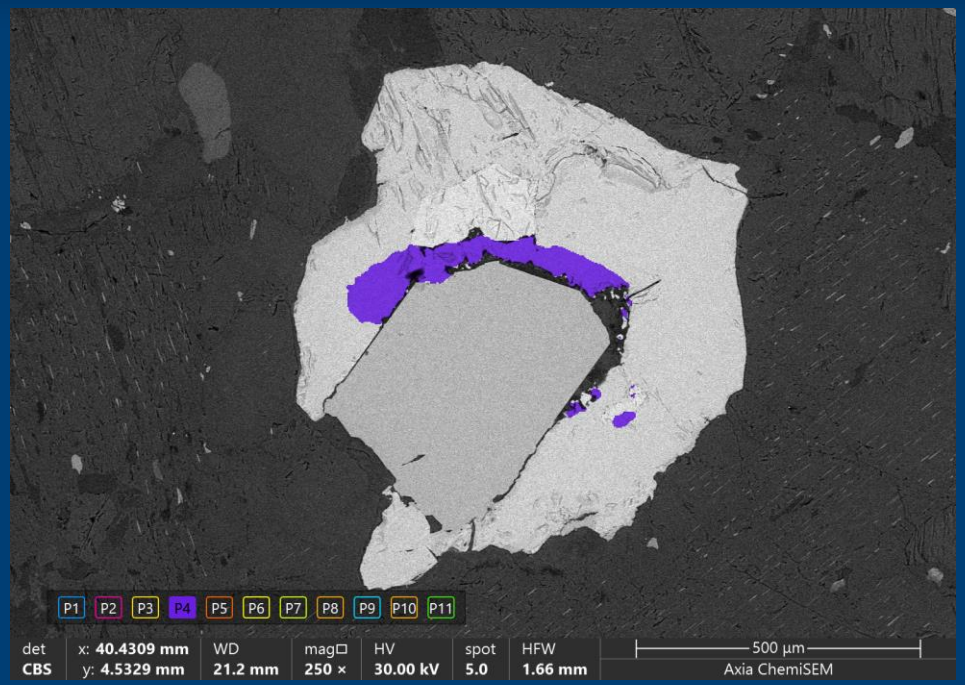

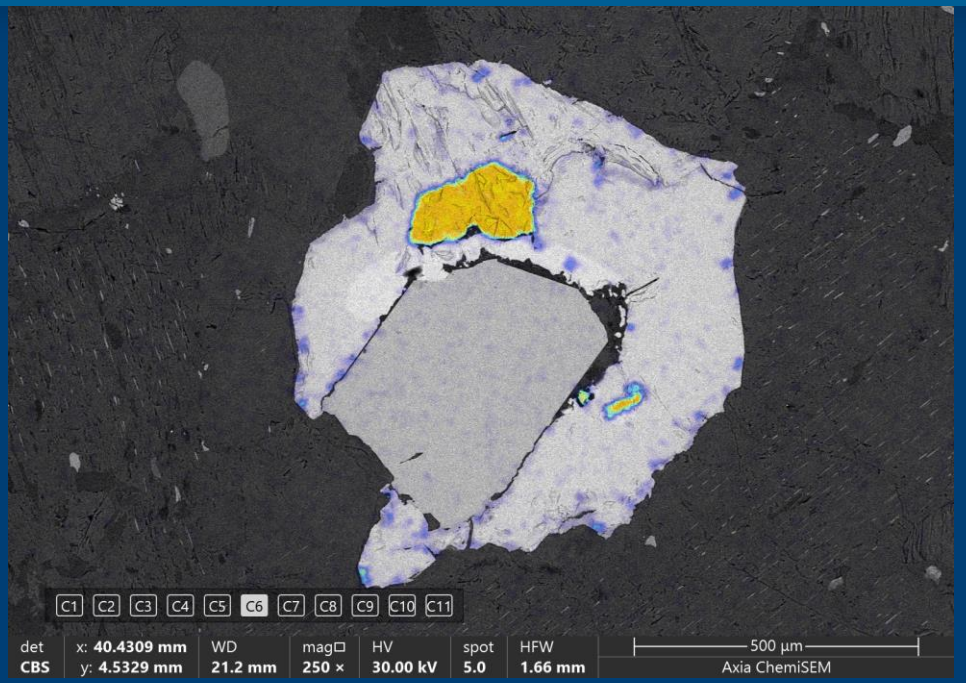

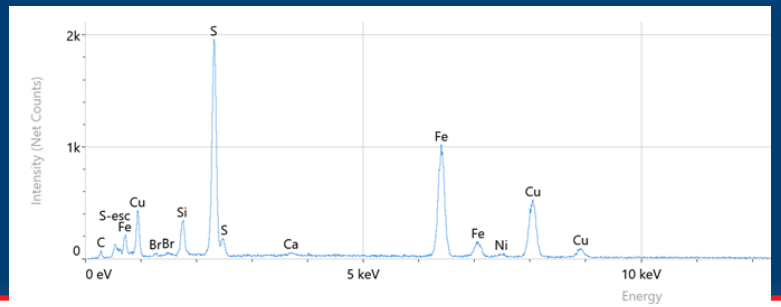

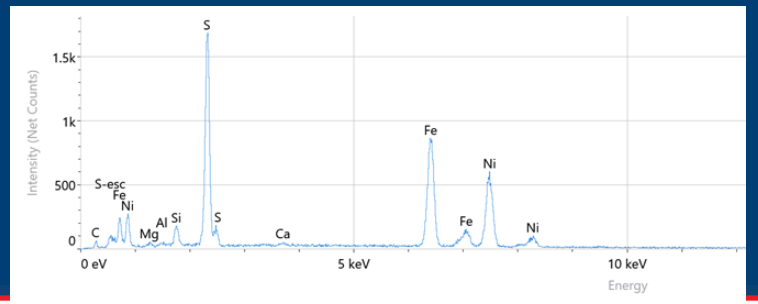

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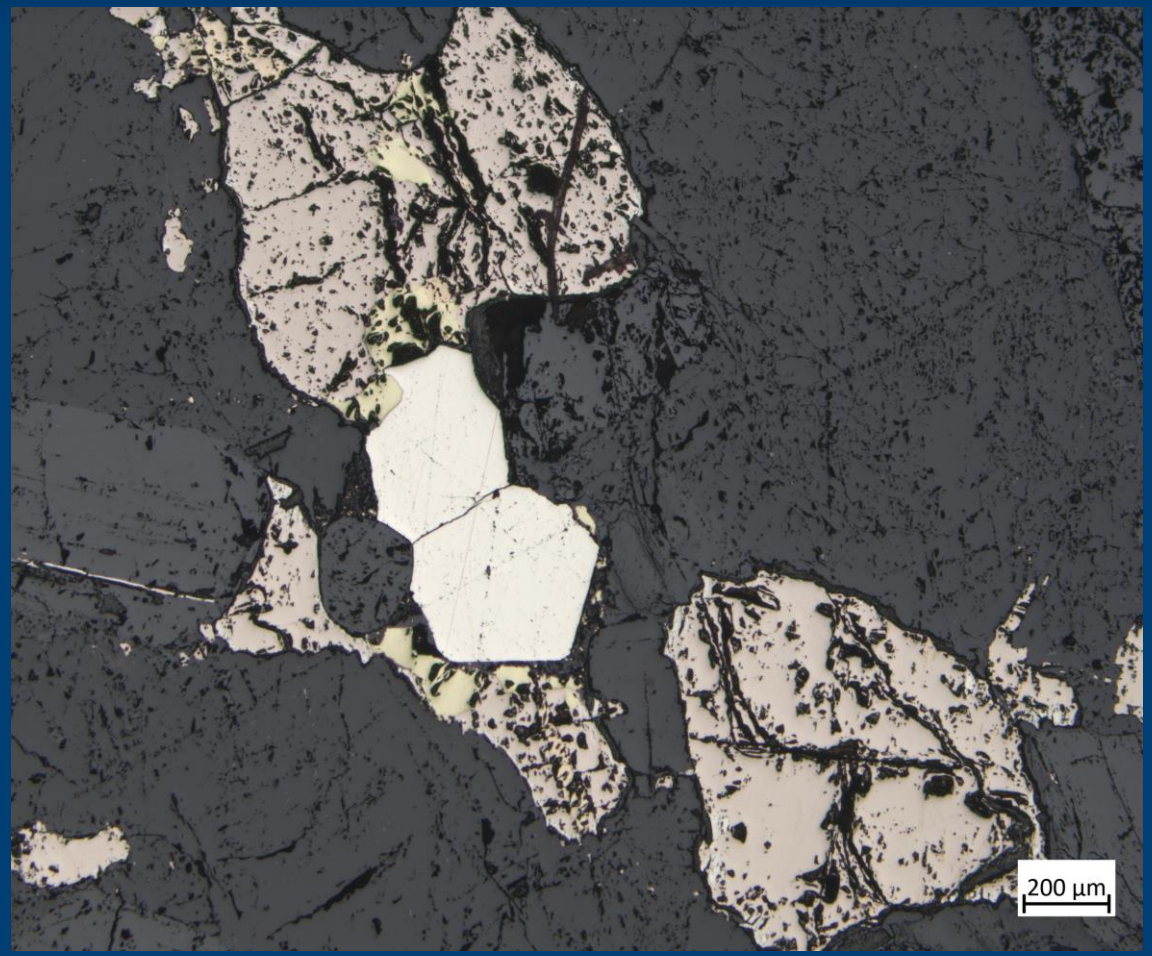
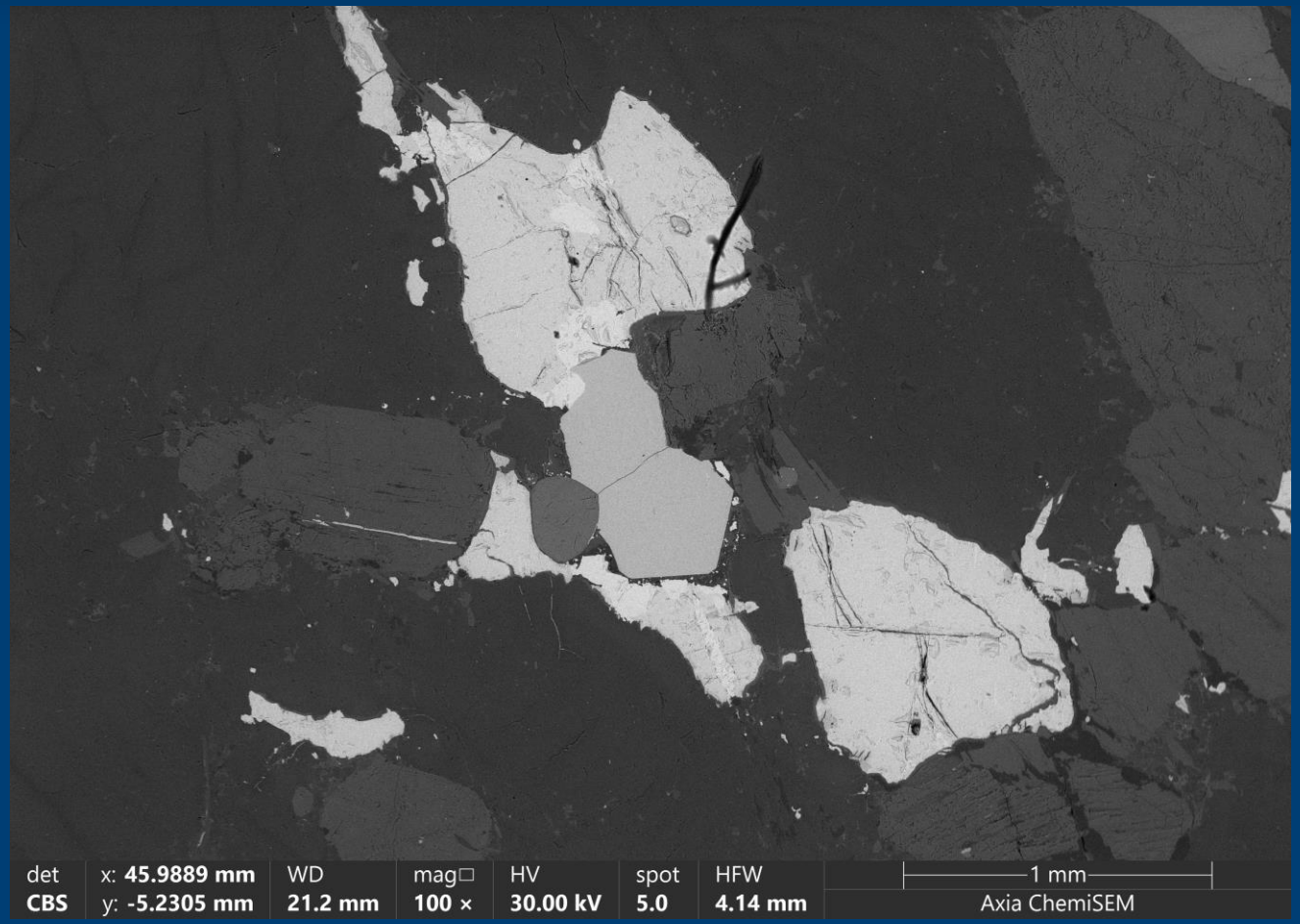
SCANNING ELECTRON MICROSCOPY





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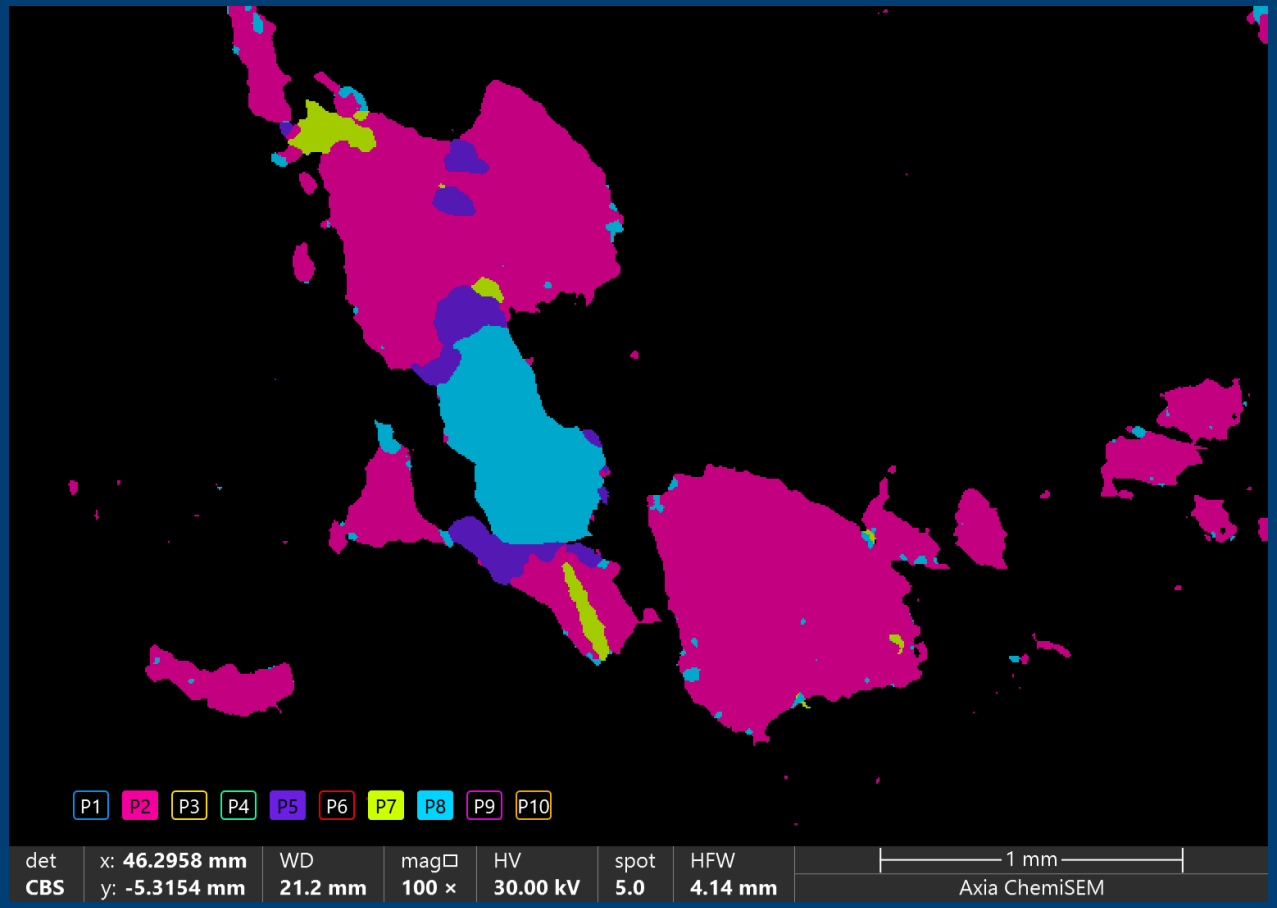
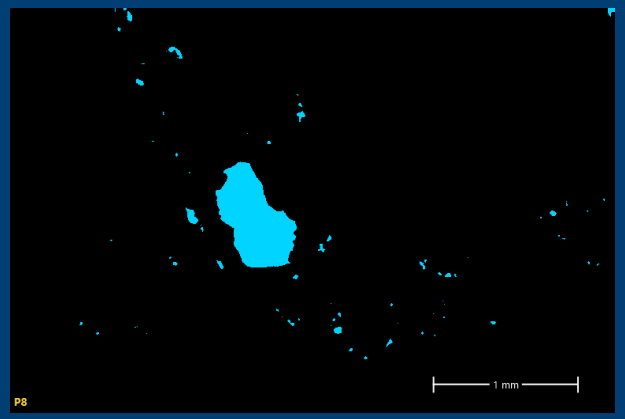
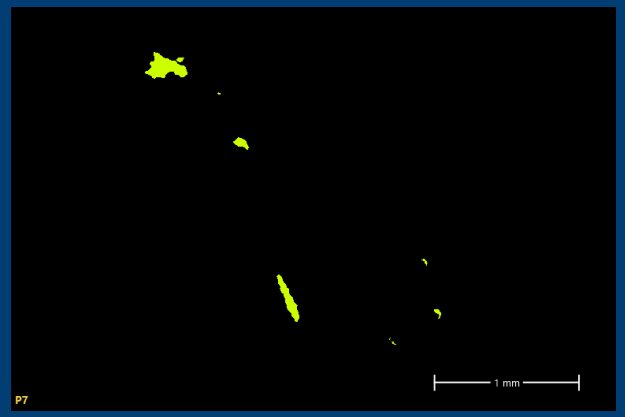
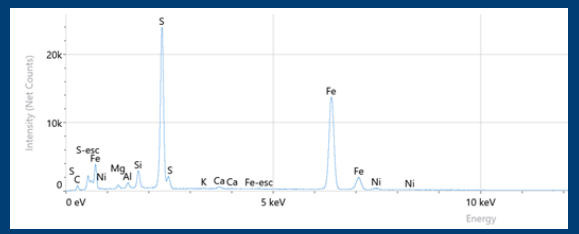


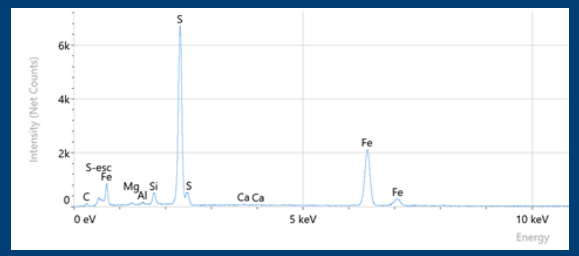
Name	Color	Area (µm²)	Area (px)	Area %
Phase 2		1514417.6	52 015	77
Phase 5		107609.1	3 696	5
Phase 7		53921.0	1 852	3
Phase 8		282415.7	9 700	15

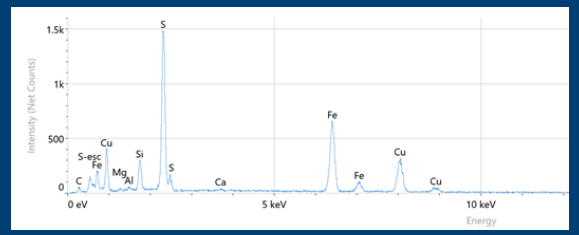
EDS X-RAY PHASE MAPPING

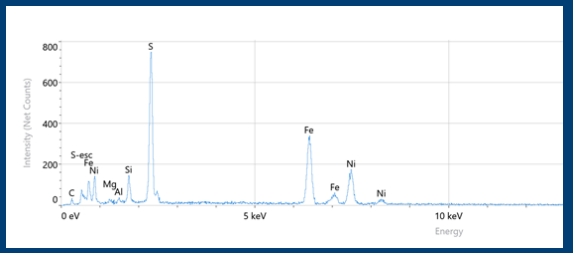












DATA COMPILATION –

RELATIVE ABUNDANCE %

GRAIN	PYRITE	PYRRHOTITE	CHALCOPYRITE	PENTLANDITE
1	29	70	6	5
2	15	77	5	3
3	20	65	8	7
4	24	72	4	0
AVG	**22**	**71**	**23**	**4**

- **USE TOTAL SULFUR VALUES TO CALCULATE THE WT% OF EACH MINERAL**
- **TABULATE % OF GRAINS IN EACH SIZE FRACTION CONTAINING IRON SULFIDES**
- **USE IN C295 CALCULATIONS**

QUESTIONS

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