Solid-state synthesis of boracites

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Solid State Synthesis of Boracites

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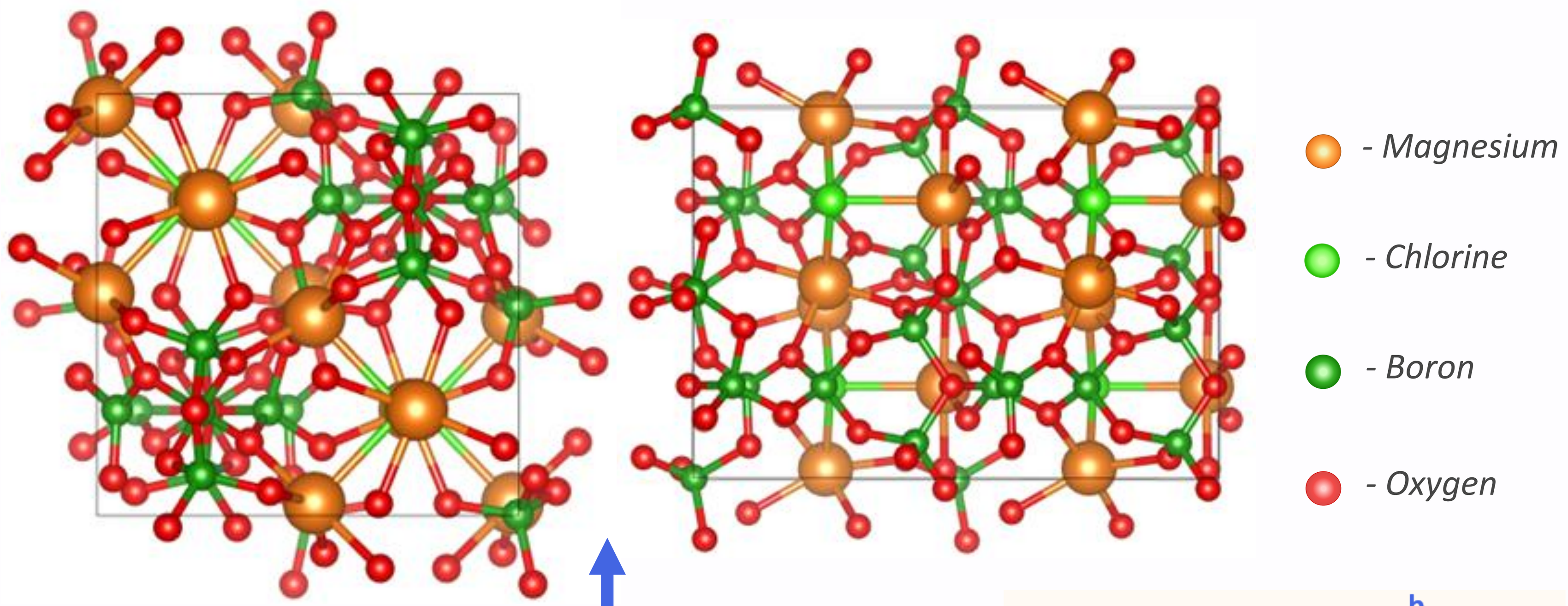
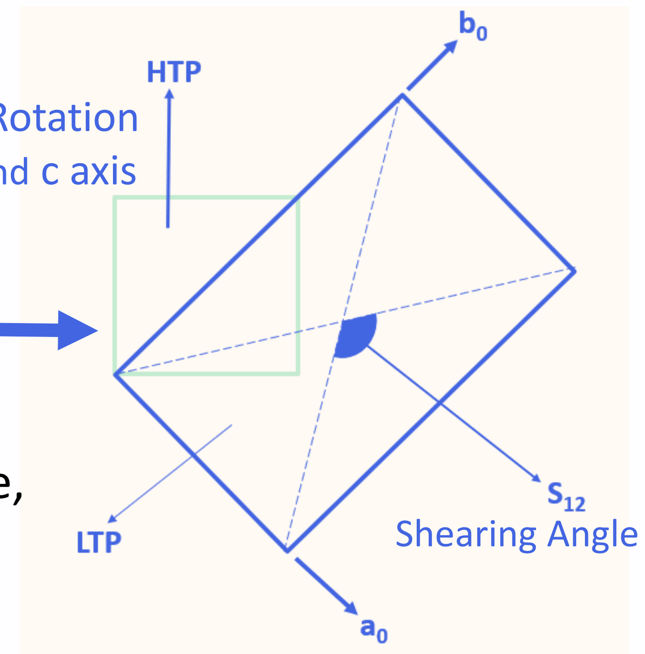
Introduction

Boracites of general formulas, M3B7O13X (M = transition metal & X = halogen) are a family of material with the same structure as the natural minerals.



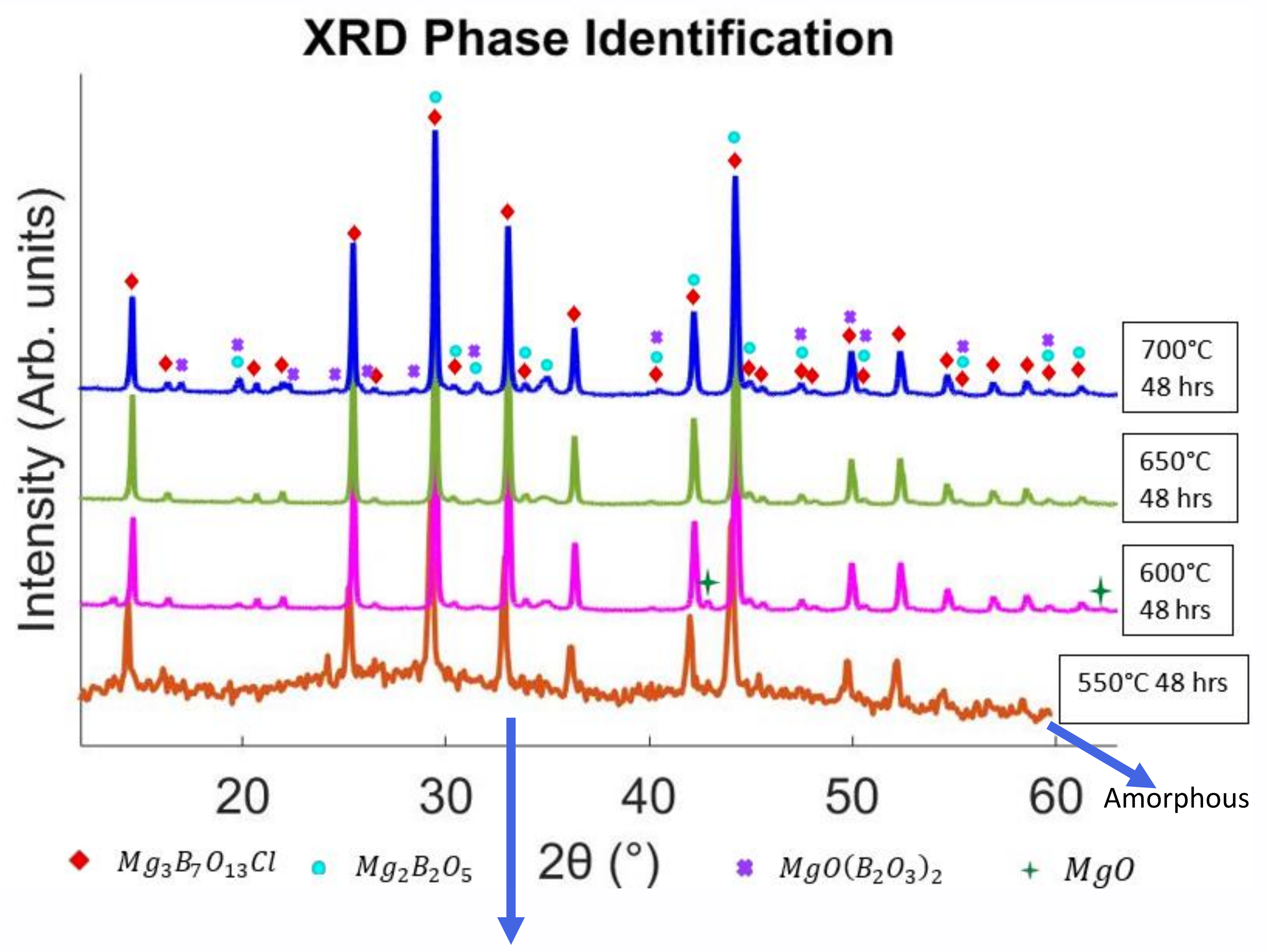
General structure of MgCl boracite.



They are improper ferroelectrics that undergo a shearing during their cubic-orthorhombic phase transitions.

Negative capacitance was observed in Cu-Cl boracite, as Mg and Cu have the same ionic radius we aim to look at whether Mg-Cl show the same electrical behaviour.

Phase Identification



Most prominent secondary phase is $Mg_2B_2O_5$ because of the volatility of chlorine.

Solid State Synthesis

$$3Cl_2Mg \cdot 6H_2O + 7H_3BO_3 \rightarrow Mg_3B_7O_{13}Cl + 5HCl + 26H_2O$$

I prepared different methods of solid-state synthesis exploring different temperatures and times.

Agate pestle & mortar

Reagent mixture ground

FURNACE

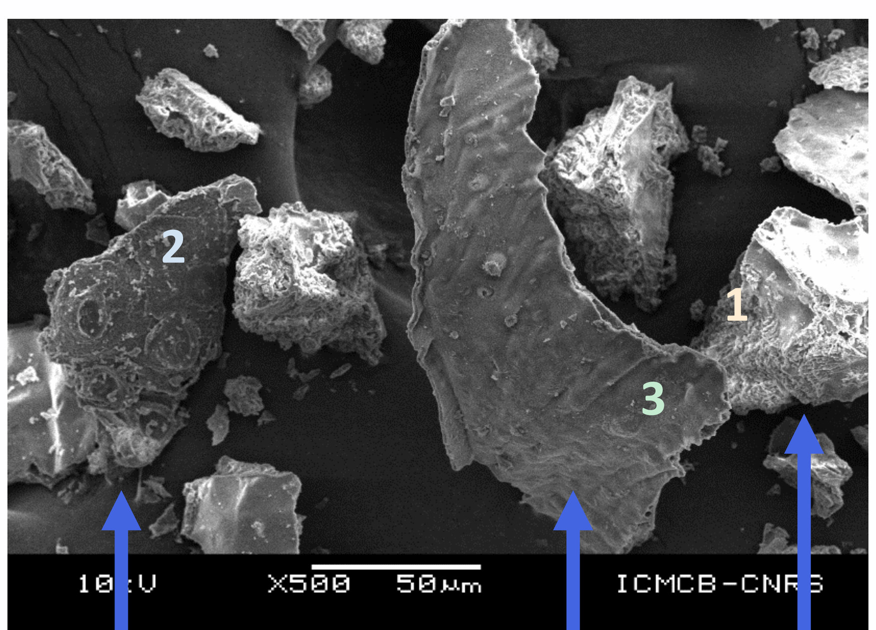
650˚C /48 hrs

Consistent calcination between 550°C – 700°C.

Refinements

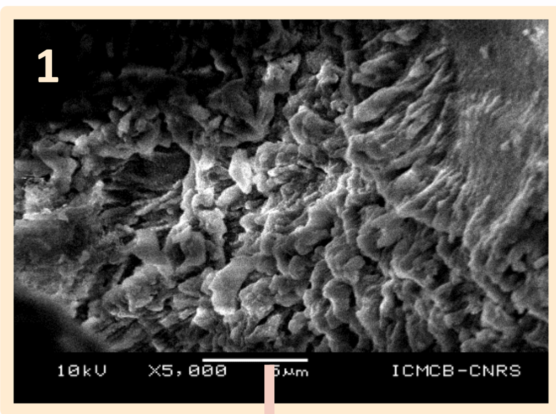
Sample Conditions	Unit Cell Parameters			Shearing Angle	MgCl Boracite Phase %	Mg Borate Phase %
	a	b	c			
600°C 48hrs	8.553(3) Å	8.543(2) Å	12.109(1) Å	0.0342(3) °	90.4(6) %	9.6(6) %
650°C 48hrs	8.548(2) Å	8.539(2) Å	12.100(3) Å	0.0328(2) °	89.3(4) %	10.7(4) %
700°C 48hrs	8.5492) Å	8.537(2) Å	12.100(4) Å	0.0391(1) °	84.1(5) %	15.9(5) %

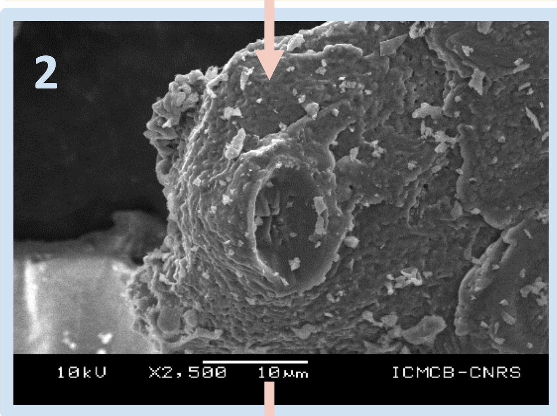
Three Phase Morphology

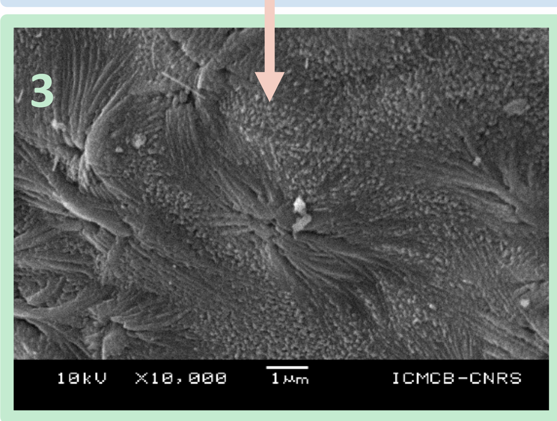


The three morphologies appear to be different stages of each other that are developing from one another. This agrees with the similarity in structure between boracites and borates.

Three distinct morphologies = three phases







Most likely to be the MgCl boracite, seems to be the final stage of the morphology progression.

Conclusions

Shearing angle isn't affected by the calcination conditions, the secondary phases or therefore the volatility of the chlorine.

Consistent calcination with 84-90% purity.

Optimal calcination temperature ~600-650°C (amorphous below, volatility of Cl above).

Further effort to increase purity.

Acknowledgements

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