



CUPID Management and Chinese CUPID Groups Meeting

Research Progress on the Preparation of LMO Single Crystals at Ningbo University

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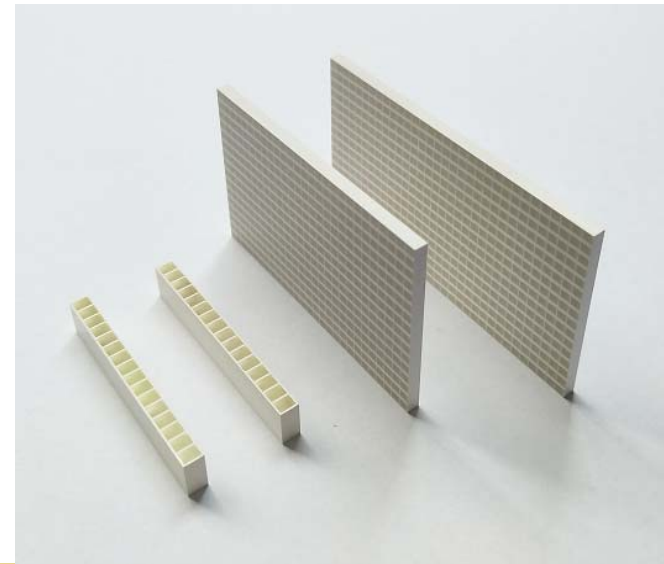


Outline

- 1 Introduction of Crystal Laboratory in Ningbo University**
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1 Introduction of Crystal Laboratory in Ningbo University

Ningbo University is a well-known university located in **Zhejiang Province, P. R. China**. The crystal materials laboratory has been focused on single crystals and their application. Several growing technique together with their equipment have been built up in our laboratory. We have developed growing techniques to prepare many crystals such as scintillation crystals, piezoelectric crystals and other optical crystals.



A series of crystal materials developed in our laboratory

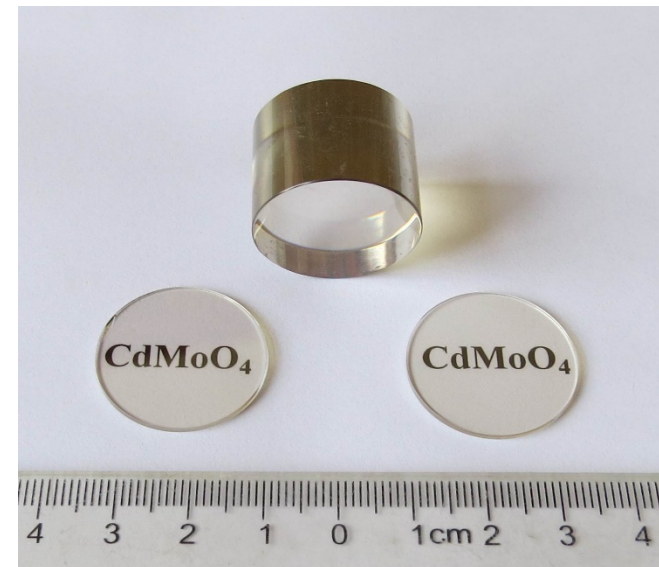
1 Introduction of Crystal Laboratory in Ningbo University



A series of crystal furnaces built up in our laboratory

2 Early Research on Molybdate crystals

- Since 2016, some Molybdate single crystals have been grown in our Laboratory:
 1. Polycrystalline powder was obtained by solid state reaction;
 2. Crystals were grown by the Vertical Bridgman method



Ø44 × 45mm

CdMoO₄ crystal boule

3 Vertical Bridgman Growth of LMO Single Crystals

Polycrystalline material preparation



Crucible fabrication



Feed material filling



Crucible sealing



Assembling into furnace

Crystal growth



Crystal boules



Crystal orientation



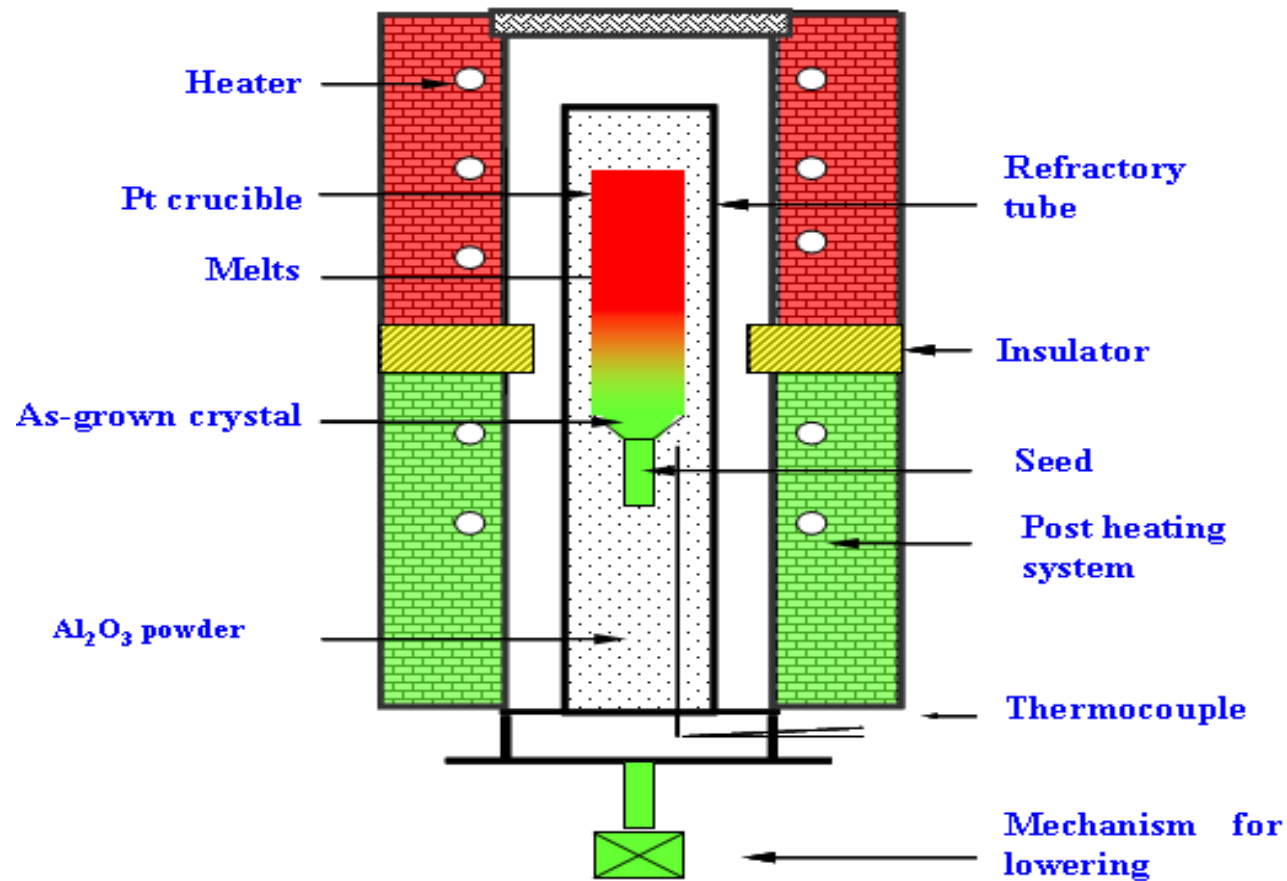
Crystal fabrication



Crystal elements

The technical process of the Vertical Bridgman growth of LMO single crystals

3 Vertical Bridgman Growth of LMO Single Crystals

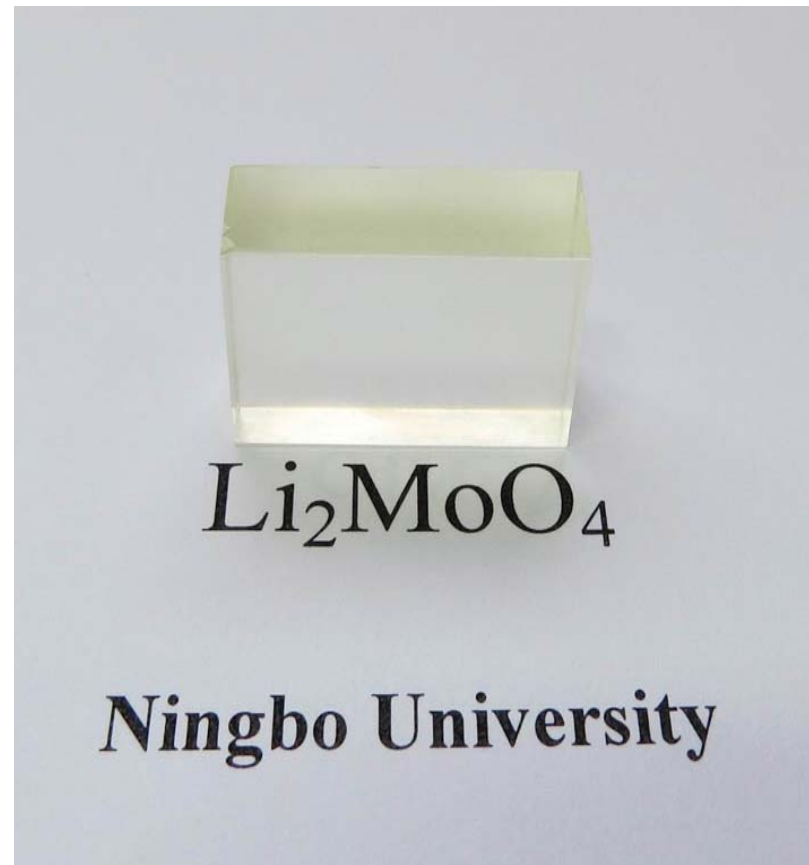


Schematic illustration of the Vertical Bridgman furnace

3 Vertical Bridgman Growth of LMO Single Crystals



Bridgman furnace in our laboratory



**LMO single crystal grown
by Bridgman method**

4 Czochralski Growth of LMO Single Crystals

Polycrystalline material preparation



Zone-melting purification



Crystal growth by CZ technique



Crystal boule

Annealing treatment



Crystal fabrication



Crystal polishing



Crystal elements

The technical process of the Vertical Bridgman growth of LMO single crystals

4 Czochralski Growth of LMO Single Crystals



Czochralski furnace in our laboratory

4 Czochralski Growth of LMO Single Crystals



**$\text{\O}65\text{-}75 \times 130 \text{ mm}^3$ LMO crystal boule grown in our laboratory
(Using unrefined raw materials)**

4 Czochralski Growth of LMO Single Crystals



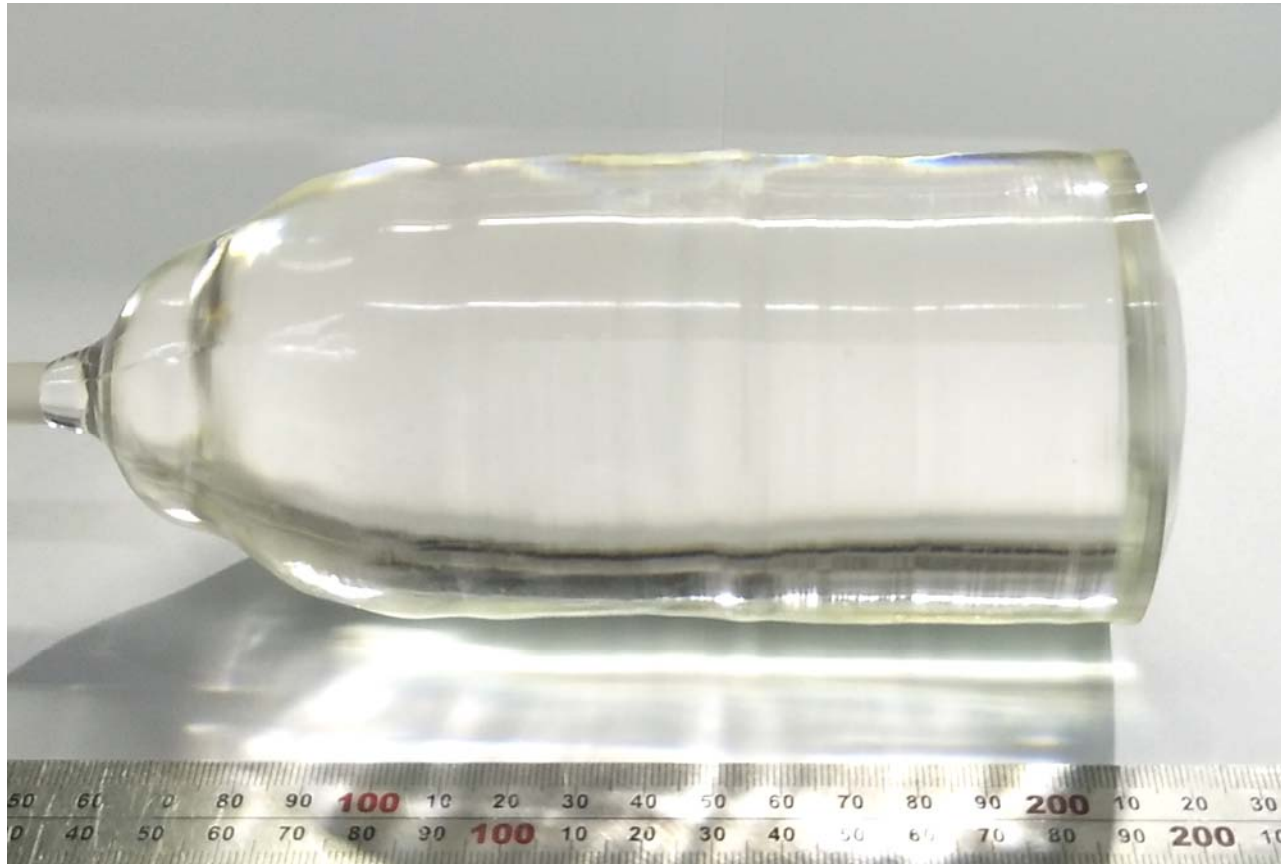
**Ø68-75 × 130 mm³ LMO crystal boule grown in our laboratory
(Using refined raw materials)**

4 Czochralski Growth of LMO Single Crystals



**$\text{Ø}70\text{-}75 \times 130 \text{ mm}^3$ LMO crystal boule grown in our laboratory
(Using refined raw materials)**

4 Czochralski Growth of LMO Single Crystals



**$\text{Ø}70\text{-}75 \times 130 \text{ mm}^3$ LMO crystal boule grown in our laboratory
(Using refined raw materials)**

4 Czochralski Growth of LMO Single Crystals



$\text{Ø}70\text{-}75 \times 130 \text{ mm}^3$ LMO crystal boule
(Ningbo University)



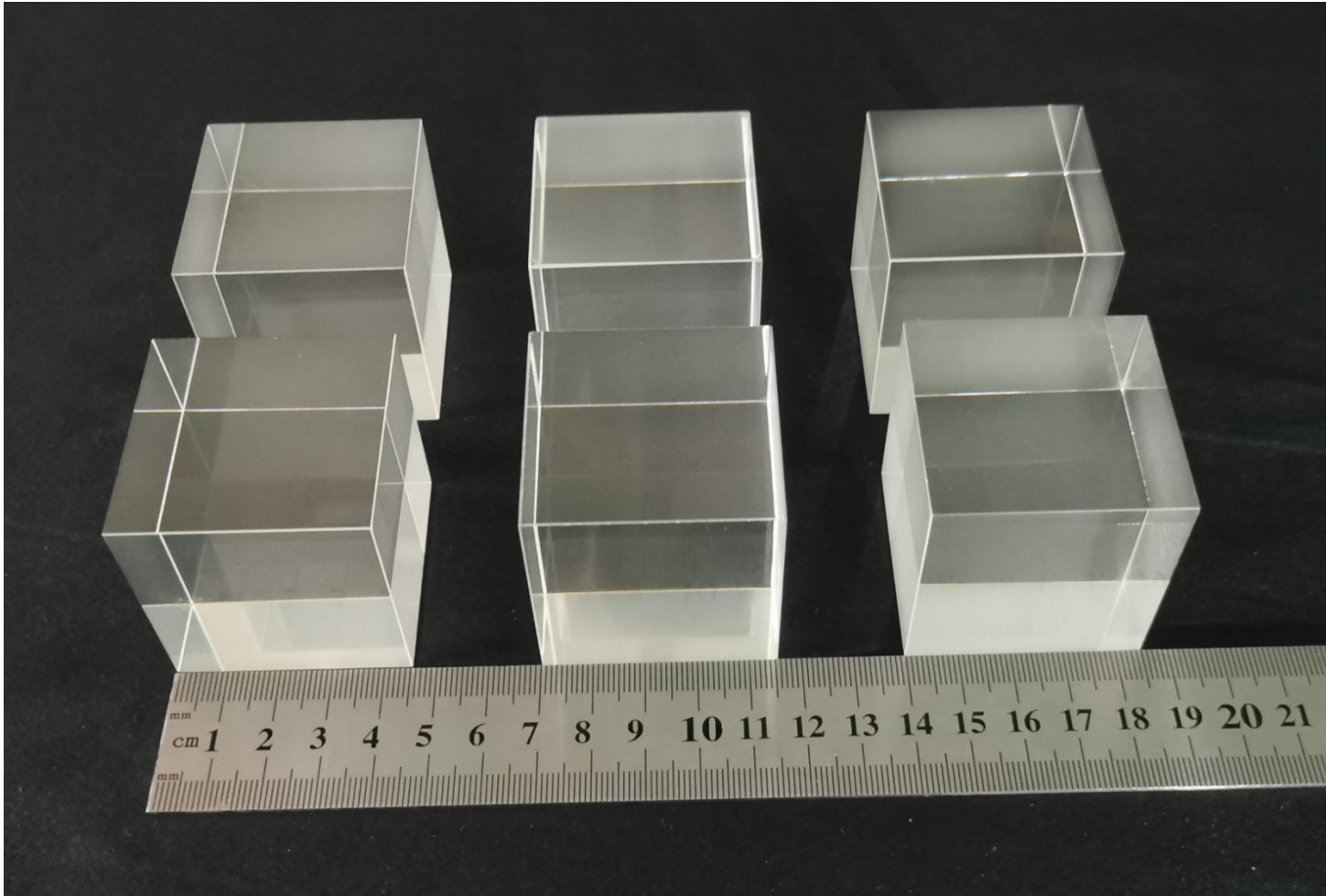
LMO crystal boule
(Russian Academy of Sciences, Siberian Branch)

5 Optical Processing of LMO Single Crystals



Unpolished LMO cubic crystal elements
($45 \times 45 \times 45 \text{ mm}^3$)

5 Optical Processing of LMO Single Crystals



**Polished LMO cubic crystal elements prepared in our laboratory
($45 \times 45 \times 45 \text{ mm}^3$)**

6 Impurities Analysis

ICP-MS analysis results

(Shanghai Institute of Applied Physics (SINAP), Chinese Academy of Sciences)

Sample	Na	Mg	Al	Ca	V	Cr	Mn	Fe	Co	Ni	Cu	Ga	Rb	Ag
$\mu\text{g/kg}$														
LMO-1	< 33	< 7	< 11	< 83	< 9	< 4.2	10.3	< 1161	6.09	< 28	< 4.5	1.02	239	1.61
LMO-2	< 33	< 7	< 11	< 83	60.3	15.7	< 2.4	< 1161	0.097	< 28	< 4.5	0.193	1.35	3.87

Sample	Sn	Te	Cs	Ba	Pt	Tl	Pb	Bi	K	W	^{232}Th	^{238}U
$\mu\text{g/kg}$										mg/kg	ng/kg	
LMO-1	2.64	97.3	1607	133	< 0.1	133	< 0.1	< 0.3	< 100	246	223	1500
LMO-2	5.71	118	32.5	109	< 0.1	109	< 0.1	< 0.3	< 100	256	73.1	331

LMO-1: initial powder

LMO-2: the small pieces of LMO crystals for growth which was refined using zone melting method

7 Conclusions and Outlook

- **Using the Czochralski technique process developed in our laboratory, high quality LMO single crystals have been prepared successfully from the refined Li_2MoO_4 polycrystalline material.**
- **The impurities in the raw materials can be reduced by the zone melting method. The crystal boules obtained are optical colorless clear.**
- **The thermal stress can be reduced by annealing the crystal boule under air atmosphere, which will be helpful to be cut without cracks.**
- **The crystal elements with the dimension of $45 \times 45 \times 45 \text{ mm}^3$ can be fabricated using the appropriate process developed in our former work.**

7 Conclusions and Outlook

- **R&D condition at NBU:**
 - a) **Twenty two vertical Bridgman furnaces can be reformed to purify the raw materials.**
 - b) **Five Czochralski furnaces can be used to grow the LMO crystals, but four furnaces should be reformed with diameter automatic controlling system.**
 - c) **20kg Pt are available to purify the raw materials and grow LMO single crystals in our lab now.**
 - d) **Ten muffle furnaces can be used to synthesis the raw materials and anneal the crystal boule before cutting.**
- **20~30 pieces of cubic elements may be produced in our lab in the future.**



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Thanks for your attention!

