X-ray Diffraction Studies of Structures of Lutetium Aluminum Oxide

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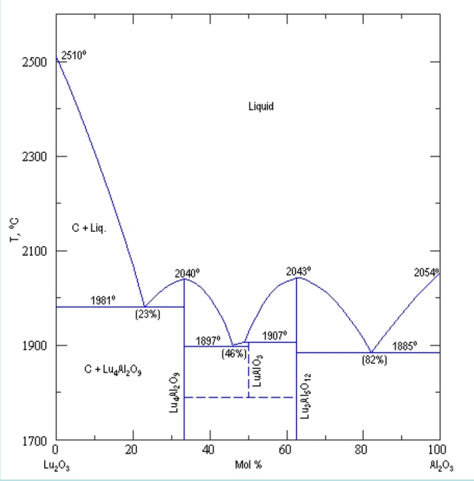
Introduction

Single crystal cerium-doped $LuAlO_3$ has the potential of being used in numerous applications including nuclear medicine, security monitoring, geophysical exploration, and non-destructive testing where efficient and fast scintillators are required. The calculated phase diagram for the Lu_2O_3-Al_2O_3 system [1] is shown below.



PDFC fig. 10396 system Lu_2O_3-Al_2O_3

In the above phase diagram three compounds are shown: $Lu_3Al_5O_{12}$ which has the garnet structure [2] (LuAG), $LuAlO_3$ which has the perovskite structure [3] (LuAP), and $Lu_4Al_2O_9$. The work here summarizes the results from five different crystal growth runs.

Experimental

Cerium-doped Lu_2AlO_3 (LuAP) was grown by the Czochralski method in inductively heated iridium crucibles in a nitrogen atmosphere to which small amounts of oxygen, ranging from 0.1 to 0.5%, was added. The starting materials were Lu_2O_3 and Al_2O_3 with a purity of at least 99.99%, with melt Ce concentrations ranging from 0.4 to 1 atomic percent. The residual melt is the fraction of the initial charge retained in the crucible after the growth run is complete.

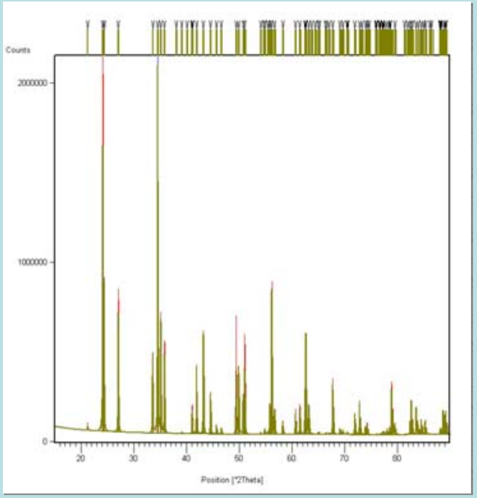
The samples were ground in an agate mortar pestle and screened down to <32 µm (450 mesh). The powder samples were contained in deep welled sample holders for data collection. X-ray powder diffraction data were collected on a PANalytical MPD X'Pert PRO θ-θ diffractometer with an X'Celerator Real-Time Multiple Strip detector using CuKα radiation. Data were collected between 15 and 90° two-theta. The scan speed was either 1700 s/step or 200s/step and is given with the results in Table 1.

The data were refined using the PANalytical Rietveld package Highscore Plus.

Table 1. Data collection conditions, phase(s) present, and physical appearance

run	Rate (s/step)	Sample ID	Phase(s) present	color
1	1700	single crystal	LuAP	colorless
	200	residual	LuAP	white (powder)
2	200	single crystal	LuAG+ $Lu_4Al_2O_9$	yellow
	200	residual	LuAG + Lu_4Al2O_9	yellow (powder)
3	1700	single crystal	LuAP	colorless
	200	residual	LuAP	white (powder)
4	1700	single crystal	LuAP	colorless
	200	residual	LuAG + $Lu_4Al_2O_9$	yellow (powder)
5	200	residual	LuAG, Lu_2O_3, and $Lu_4Al_2O_9$	pink (powder)

Results



Rietveld refinements on the data collected on the powdered samples from the colorless single crystals showed single phase $LuAlO_3$. To model the x-ray powder diffraction pattern the atomic positions for $YAlO_3$ [4] were used. The slow rate of data collection reduced the noise so that $Lu_3Al_5O_{12}$ could have been detected if present at approximately 0.2 wt% or greater. Both $Lu_3Al_5O_{12}$ and another phase, most like $Lu_4Al_2O_9$, were observed in the data collected on the samples that were yellow in color.

Attempts to model $Lu_4Al_2O_9$ as a secondary phase in the powder pattern using the atomic coordinates from $Y_4Al_2O_9$ were unsatisfactory. One crystal growth run resulted in a pink residual powder and the x-ray diffraction data revealed three phases (Lu_2O_3, $Lu_4Al_2O_9$, and $LuAlO_3$).

References

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