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Rare earth hexaborides: large single crystals

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Abstract

Single crystals of the rare earth hexaborides have been grown successfully by the floating zone technique using a high-power Xenon arc Image furnace. Large, high-quality crystals up to 1 cc in volume have been obtained for several rare earth hexaborides by this method. Crystals of these compounds have also been grown using enriched ^{11}B isotope for use in neutron scattering studies. The crystal growth and characterisation of these crystals are reported in the paper.

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Keywords: Floating zone technique; Rare earth compounds; Hexaborides; Crystal growth

1. Introduction

Rare earth hexaborides exhibit a whole range of interesting magnetic properties. They are also useful in many applications, LaB_6 crystals for example, are widely used as electron sources where good-quality crystals are essential for optimum performance. Materials such as borides in general have very high melting points ($\sim 2400-3000°C$) which often poses a serious problem in devising methods to obtain good-quality single crystals. Single crystals of the rare earth hexaborides have been grown in the past from melt using excess aluminium as flux [1]. Crystals grown by the flux method tend to be rather small, the edge lengths typically measuring a few millimeters. Several of the rare earth hexaborides have congruent melting points and therefore may be grown with ease by the floating zone technique. The crystal growth of these materials has been previously carried out by this technique, using RF power to melt the materials, due to their high melting points [2,3]. This paper reports the growth of large, high quality single crystals of a series of rare earth hexaborides using a high-power image furnace equipped with Xenon arc lamps.

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2. Experimental procedure

Commercial rare earth hexaboride (RB_6) powders from Cerac, USA (99.9% pure) were used as starting materials. The powders containing ^{11}B isotope were prepared by reacting high-purity rare earth oxides and ^{11}B isotope powder (99.5% enrichment, Eagle Picher, USA) in a hot press at 1700°C under vacuum. The RB_6 powders were compressed in the form of rods and sintered in argon gas at 1550°C for an hour prior to the crystal growth. An image furnace equipped with four mirrors and 3 kW Xenon arc lamps (CSI Model FZT-1200-X-VI-VP) was used for the crystal growth. The growths were carried out by the floating zone method in a flow of argon gas at growth speeds of $10-18$ mm/h with the feed and seed rods rotating at 30 rpm. The crystal boules produced were examined using X-ray Laue photography and compositional analysis was done using an electron microscope.

3. Results and discussion

Rare earth hexaborides for $R = La, Ce, Pr, Nd$ have congruent melting points and crystals of all these were grown by the method described. The boules obtained were roughly 6 mm in diameter and lengths varied from 40 to 70 mm. The boules were colourful, ranging from a

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Fig. 1. Photograph of an as-grown boule of CeB_6, grown with enriched ^{11}B isotope for use in neutron scattering studies.

bright pinkish purple in the case of LaB_6 to a deep bluish purple in the case of NdB_6. The growth conditions were identical in all cases and the molten zone was stable for the entire growth period. The growth of the crystals containing the ^{11}B isotope posed no additional problems. The crystals grown in the first growth were used as seed crystals to provide the orientation for all subsequent growths. Fig. 1 shows the photograph of an as-grown boule of CeB_6, with the seed still intact.

In spite of the large temperature gradient that the growing crystal is subjected to in this method of growth, the boules are crack-free and of good quality. The as-grown boules show remarkably good X-ray Laue diffraction patterns without the need for any polishing. All the crystals pick up one of the principal axes as their growth axis quite easily. Spark erosion was used to cut slices off the grown boule for tests and measurements. Where the crystal cleaved, shiny, sparkling surfaces are obtained.

The crystals grown with enriched ^{11}B isotope are for use in neutron scattering experiments that are planned in the future. The method described here provides the ideal route for obtaining samples for these experiments for which large volumes of high-quality crystals are required. Unlike the crystals grown using Al flux, the crystals grown using the image furnace do not suffer from any inclusions or contamination.

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