and increased incorporation of Cr ions both contribute to subnormal effective internal magnetic fields. In summary, starting with a solid solution of iron ions incorporated in a Cr_2O_3 lattice (sample 1), increasing Fe content results in the formation of super paramagnetic clusters of iron ions which are initially held on the surfaces of chromia hosts with a low symmetry of crystal field around them still resulting in the doublet character of Mössbauer spectra (sample 2). With further increase in the iron oxide component, the iron ion clusters grow in size and develop crystallinity although retaining, to some extent, the inhibiting effects due to chromium ions in the α-Fe_2O_3 lattice, as has been shown by magnetically ordered spectra with subtle diminutions in H_n values.

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X-ray topographic assessment of flux-grown crystals of rare earth germanates ($R_2Ge_2O_7$)

The growth of single crystals of the tetragonal rare earth germanates from a flux of R_2O_3, PbO, PbO_2, GeO_2 and PbF_2 was first reported by one of us in 1973 [1]. In subsequent experiments, the flux was modified to contain a large excess of GeO_2, and MoO_3 was added as a major component. With this composition, it was possible to reduce flux evaporation and to allow crystals to grow by slowly cooling the melt. Large, optically clear, equidimensional crystals of $R_2Ge_2O_7$ were obtained, up to 3 mm x 3 mm x 3 mm, and basal plates up to 5 mm x 5 mm x 1 mm thick, some of which were free from visible inclusions [2]. In this communication, the study of a substantial number of such platelets by X-ray topography is briefly reported.

As-grown crystals of plate-like habit, leached from the flux, were selected on the basis of optical perfection and were examined without cutting or polishing. For $AgK\alpha_1$ radiation, the product of absorption coefficient and thickness, μt, was typically 2 to 10.

All the crystals examined were bent. Only the crystal shown in Fig. 1 was sufficiently uniform for Lang topographs to be taken, and here only one reflection could be obtained. Lang topographs, which rely on collimated characteristic radiation, could not be taken of the others and, because the bending was about two orthogonal axes, a Bragg angle controller proved little use. Very rapid

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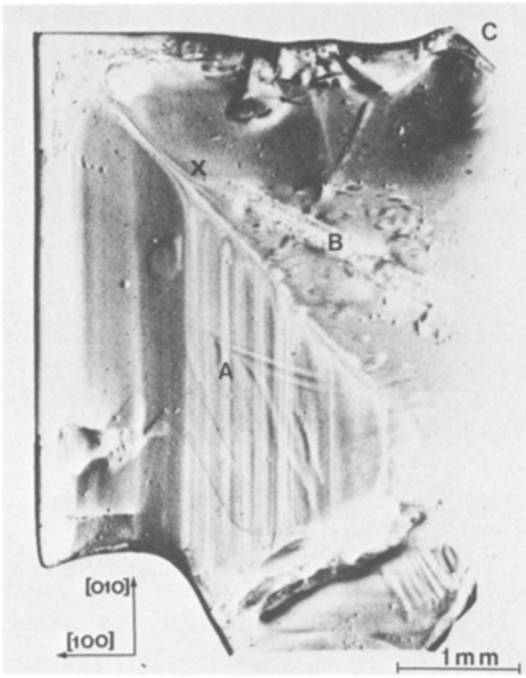

Figure 1 Lang-type X-ray topograph of $Tb_2Ge_2O_7$ crystal, 4 0 0 reflection, $AgK\alpha_1$ radiation.

assessment was, however, possible by means of synchrotron radiation for X-ray topography [3], which exploits the continuous nature of the synchrotron radiation. Each point on the crystal selects its own wavelength for Bragg reflection and complete topographs can be obtained. In addition, many reflections are recorded simultaneously and in a matter of seconds. Use of synchrotron X-ray topography (SXRT) enabled us to assess very rapidly the perfection of many crystals. Despite the bending, the crystals were remarkably free from lattice defects.

Fig. 1 shows a Lang topograph of a 200 μm thick crystal of $Tb_2Ge_2O_7$. Under the high absorption conditions ($\mu t \simeq 4$) the presence of "anomalously" transmitted intensity is itself a good indication of high lattice perfection. Strong growth bands are visible (A), demonstrating that substantial fluctuations in lattice parameter took place, presumably due to variation in impurity content caused by convectional movement of the solution. The growth bands correspond to steps on the crystal surface which can be seen optically. This indicates that growth rates in the *c*-direction, at least, are dependent upon impurity concentrations. As there is only a change in lattice parameter normal to the growth front, growth bands in sector B are invisible in this particular reflection. In this crystal, growth occurred at equal rates on only two of the {1 0 0} faces, and this asymmetry presumably arose from nucleation close to the crucible wall, resulting in subsequent starving of two surfaces of solute. In very many of the crystals examined, equal growth rates were found on all four {1 0 0} surfaces, leading to perfectly rectangular crystals with a central nucleus.

Two distinct periods of growth can be distinguished in the crystal illustrated in Fig. 1. Early growth was irregular, resulting in strong growth bands and small inclusions, while later growth was much more uniform. The boundary between the two regimes is abrupt and the contrast of the growth sector boundary at this point (X) is noteworthy. Examination of the crystal under an optical microscope with light incident at an angle to the surface indicates the presence of flux inclusions at this point and along the growth sector boundary. Other flux inclusions seen optically correspond to the precipitates visible in the topographs. Growth bands can be used to trace the growth history of a crystal [4, 5] and hence the continuous curving of the growth band from

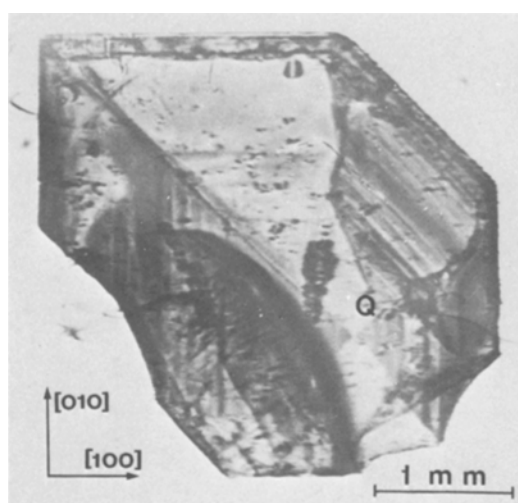

Figure 2 Synchrotron X-radiation topograph of $Tb_2Ge_2O_7$ crystal exhibiting growth on both {1 0 0} and {1 1 0}, 4 0 0 reflection.

[0 1 0] to [1 1 0] direction at X suggests simultaneous growth on $(1\,\bar{1}\,0)$ and (1 0 0) faces. A mechanism whereby this is possible is the formation of a needle parallel to [1 1 0] by rapid initial growth on (1 1 0) and subsequent infilling by growth on {1 0 0} faces. Considerable numbers of flux inclusions are present in the area of unevenness of the growth surface at C where rapid growth in the $[\bar{1}\,1\,0]$ direction had taken place.

Growth on {1 1 0} faces was, in fact, occasionally observed, as in the synchrotron X-ray topograph of the crystal of $Tb_2Ge_2O_7$, shown in Fig. 2. Here, we see that growth initially took place at equal rates on all four {1 0 0} faces. However, at point Q growth abruptly started on (1 1 0). The growth rate on this face is comparable with that on {1 0 0} and, from the non-linearity of the growth sector boundaries, we deduce that growth is energetically competitive on both {100} or {1 1 0}. Flux inclusions along the growth sector boundaries are visible optically. Very large changes in composition might lead to {1 1 0} growth becoming dominant, resulting in needle growth, as was deduced in Fig. 1. Other samples with very few, or no, visible flux inclusions have fewer surface steps and exhibit much weaker growth bands. These samples were usually thicker and in those examined, growth always occurred on {1 0 0} planes. From both Figs. 1 and 2, it appears that low impurity fluctuations, i.e. weak growth banding, favours growth on {1 0 0}, while increasing fluctuation, with the formation of precipitates, favours growth on {1 1 0}. These observations are in accord with evidence that the growth rate on various planes in fluorite is very sensitive to impurity concentration [4].

It is possible that the two curved lines in Fig. 1 might be dislocations, but apart from this tenuous identification, no dislocations were observed in any of the crystals examined. The large unit cell, and hence Burgers vectors, in this structure imply large elastic line energy, and it appears that no growth accident was sufficiently energetic to favour the nucleation of dislocations. Once again [6], growth by slow cooling of a fluxed melt, relying on spontaneous nucleation, has resulted in highly perfect crystals of an exotic compound.

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