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X-ray observation of recrystallization in deformed, 99.2% pure ytterbium

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The behaviour of the rare earth metal ytterbium at temperatures above 300 K have been investigated by several workers [1–11]. It has been established that ytterbium has a predominant face centred cubic structure, β(f c c), at 300 K and atmospheric pressure. On heating, the β(f c c) structure transforms initially to a hexagonal close packed, α'(h c p), structure in the temperature range 533 to 660 K [3, 6, 8, 11]. On further heating, the transformation to a body centred cubic structure, γ(b c c), is observed in the temperature range 970 to 1071 K [6–9]. In this letter we report that on heating 99.2% pure, deformed ytterbium, in addition to the structural transformations, recrystallization and grain growth also occur. Details of the impurities in the 99.2% pure sample and the extent of deformation have been described in a previous publication [11].

In the course of a systematic investigation on the high-temperature behaviour of deformed ytterbium, diffraction patterns were recorded at temperatures greater than 300 K, using the heating device described by Mani and Vijayan [12], a Philips powder diffractometer and Cu$K\alpha$ radiation. It was then observed that the intensity distribution in the diffraction pattern recorded from ytterbium at 400 K was very different from that recorded at 300 K. In particular, it was found that the most intense reflection (1 1 1) from the β(f c c) phase at 300 K was no longer the most intense reflection at 400 K. On the other hand, it was observed that the intensity of the reflection (2 0 0) had increased very anomalously at 400 K. A schematic representation of the observed intensity distribution at 300 and 400 K is given in Fig. 1a and b, respectively. In order to check whether the anomalous intensity distribution could be due to the presence of texture, back-reflection photographs were recorded in the temperature range 300 to 650 K using the *in situ* heating device fabricated for the Philips powder diffractometer [12]. Back-reflection photographs recorded at several stages of heating clearly ruled out the presence of texture. However, they led to the detection of marked changes in the grain structure of ytterbium occurring at temperatures $\geqslant$ 400 K. Fig. 2a to c shows the back-reflection photographs obtained at 300, 400 and 525 K, respectively. The smooth diffraction rings observed at 300 K (Fig. 2a) are found to persist to 395 K. However, at

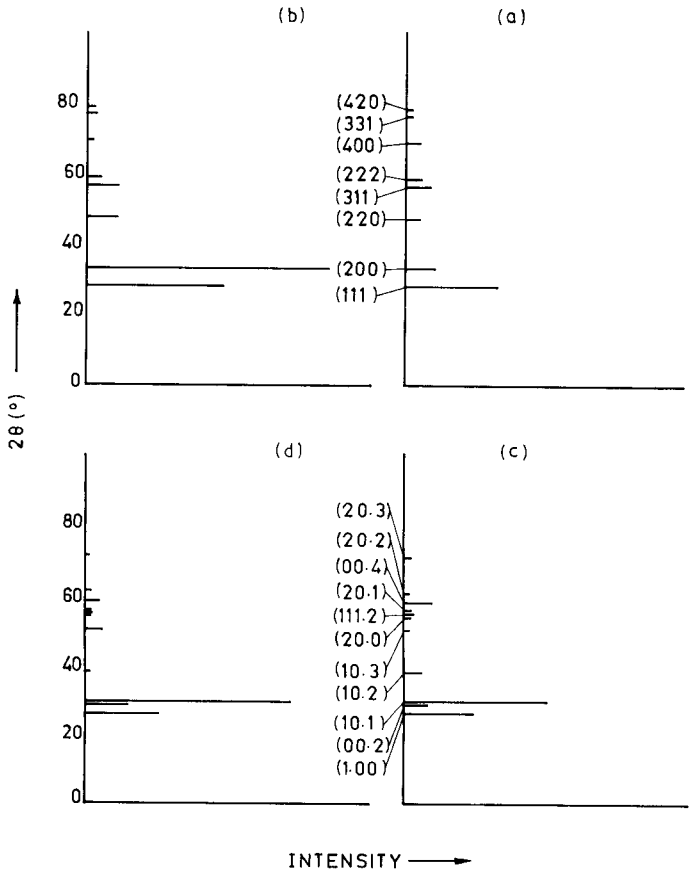

Figure 1 Schematic representation of the observed intensity distribution from deformed ytterbium at (a) 300 K, (b) 400 K, (c) 675 K, and (d) 775 K.

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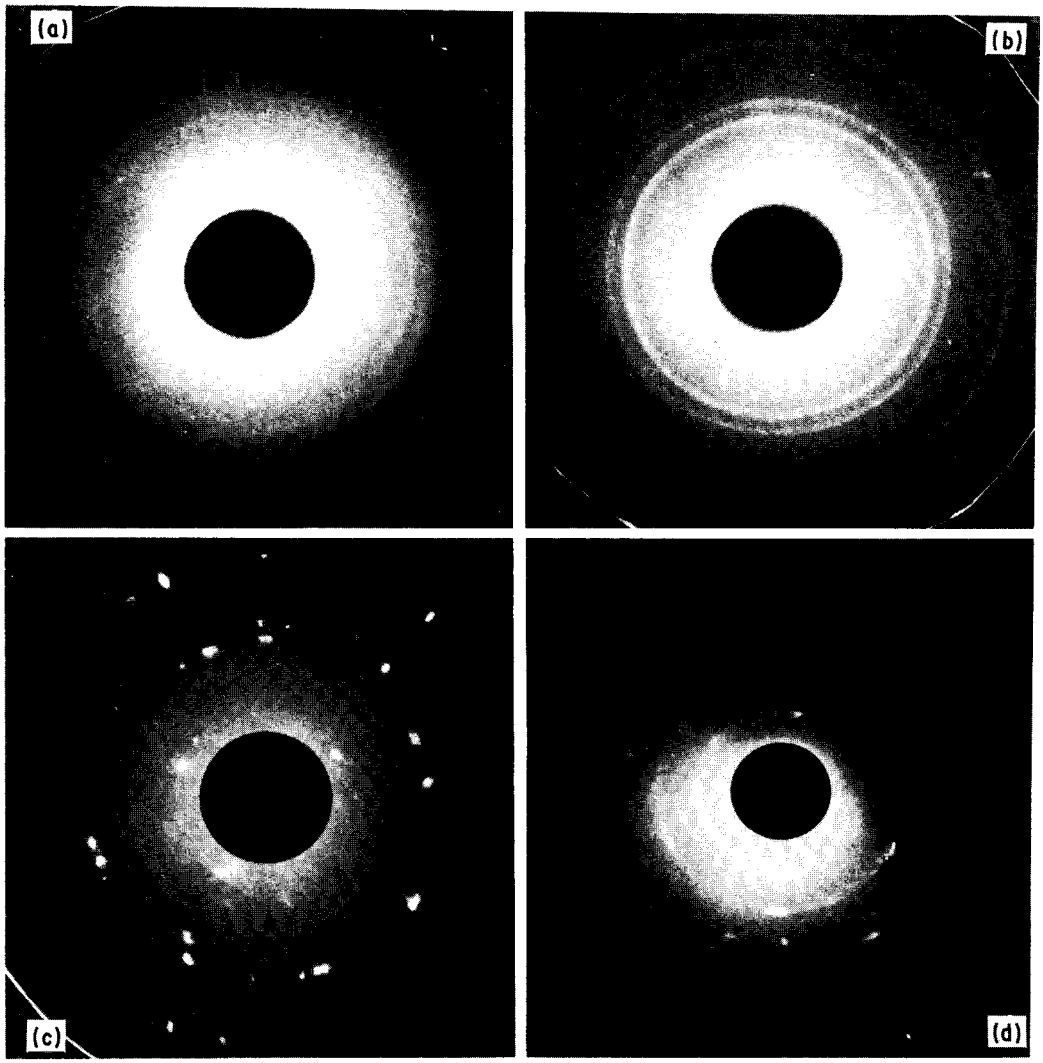

Figure 2 Back-reflection photographs from deformed ytterbium sample at (a) 300 K, (b) 400 K, (c) 525 K, and (d) 2 to 3 weeks after cooling from the α'(h c p) phase.

400 K, the diffraction rings acquired a spotty character (Fig. 2b), suggesting the onset of recrystallization. At 525 K (Fig. 2c), the pattern consisted of well-defined spots distributed along circular rings corresponding to the reflections (6 2 0), (5 3 3), (6 2 2) and (4 4 4), from the β(f c c) phase. It has been observed that increase in temperature leads to a reduction in the total number of spots in the pattern and an associated increase in the spot size (Fig. 2b, c).

These features suggest the gradual growth of grains formed at 400 K. It has been verified that the growth of grains continued in the α'(h c p) phase also. The distribution of intensities in the diffractometer patterns corresponding to the α'(h c p) phase (Fig. 1c and d) provide further support to the grain growth persisting in the α'(h c p) phase. Further, it has been observed, that on cooling the ytterbium samples from either the β(f c c) or the α'(h c p) phase, the recrystallized grains remained stable for a period of 2 to 3 weeks (Fig. 2d). Mechanical working on the sample surfaces, however, destroyed the grain structure.

Based on these observations, the anomalous intensity distribution found in Fig. 1b to d could be attributed to the recrystallization and grain growth. It is believed that whenever a large grain entered the incident beam, it led to a sharp increase in the intensity of the diffracted beam.

It may be mentioned that recrystallization in thin films of ytterbium at temperatures above the β(f c c) $\rightarrow$ α'(h c p) transformation has been reported by Gasgnier and Malaurent [10]. However, our experiments with bulkier specimens show that the recrystallization and grain growth initiate when ytterbium is predominantly in the β(f c c) phase and persist in the α'(h c p) phase also. It may be pointed out that the recrystallization temperature of 400 K observed by us is equal to $0.36T_m$, where T_m is the melting point of ytterbium. It is well known [13] that the temperature of recrystallization is related to factors such as impurity content, presence of second phase, deformation, etc. It is therefore suggested that the observed low value of the recrystallization temperature in our samples is primarily due to the presence of impurities, the extent of deformation and also, perhaps, to the size of the samples.

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