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Removal of surface strain from rare earth intermetallic compounds by ion-beam planing

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The use of argon ions to remove the surface damage introduced into brittle rare earth intermetallic compounds by lapping and polishing is described. The ion-beam planing technique allows strain-free surfaces to be prepared over large areas (~ 4 cm^2) without degradation of surface quality. The effectiveness of the technique applied to both single crystal and polycrystalline material has been demonstrated by monitoring the quality of electron channelling patterns generated from the surfaces. Microstructural detail not evident from other characterization methods has been revealed.

1. Introduction

For many magnetic and physical property measurements the ideal specimen is an oriented single crystal of well-defined quality. In the case of the rare earth intermetallic compounds, which are of particular interest because of their magnetic properties [1], assessment of crystal quality is often extremely important. For example, magnetostriction and thermal expansion behaviour in ErZn is affected by microstructural features acting as domain wall-pinning centres [2]. A variety of techniques can be used to determine crystal quality, e.g. optical metallography, scanning electron microscopy, X-ray and neutron diffraction. The use of electron diffraction or X-ray topography to identify low-angle tilt boundaries or similar microstructural defects requires thin specimens. Unfortunately thin samples of the rare earth intermetallics are difficult to prepare and handle because of their brittle nature. An alternative approach is to use electron scattering from the surface to evaluate crystal perfection: for this technique a flat strain-free surface is required. However, for most materials the preparation of polished surfaces by lapping introduces damage which both reduces the crystallographic information that can be obtained and may also limit the sensitivity of physical property measurements.

A convenient assessment of the surface strain in a sample is evident from the quality of the electron channelling pattern (ECP) obtained from the surface using scanning electron microscopy (SEM) [3]: a well-defined channelling pattern can only be produced from a flat, damage-free surface. Increases in the amount of strain cause progressive degeneration of the pattern, until ultimately no pattern is produced.

The conventional specimen preparation procedure for electron channelling studies involves mechanical polishing to produce a highly reflective and flat surface. Provided care is taken to avoid chemical attack, surface damage from lapping can often be subsequently removed by electropolishing. For many metals, polishing solutions and conditions are well known, but for most rare earth metals and rare earth-based intermetallics, polishing procedures have not been established. Since suitable solutions have to be identified for each material, the determination of new electropolishing conditions is time consuming. Ion beams are an alternative method of removing surface material. Unfortunately most commercial ion-beam systems only produce small beams (2 to 3 mm diameter) of non-uniform ion density and it is therefore not normally possible to etch large areas and maintain surface flatness. In this paper a description is given of an ion-beam technique used to remove surface damage over

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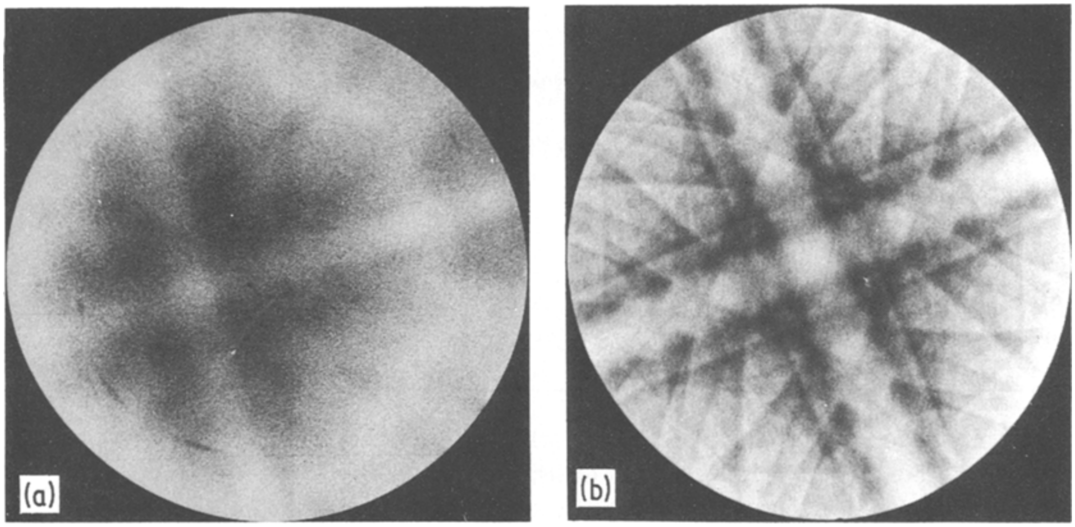

Figure 1 Selected-area channelling pattern from a $TbAl_2$ 1 0 0 single crystal, (a) after mechanically polishing down to 0.25 μm diamond paste, and (b) followed by ion-beam planing for 10 h. (Accelerating voltage 30 kV.)

large areas from some rare earth intermetallic compounds without deterioration in surface quality. The efficiency of the ion-beam planing is demonstrated by the improvement in quality of electron channelling patterns obtained from the specimen surface.

2. Experimental techniques

Single crystals of RAl_2 compounds ($R \equiv$ Tb, Gd) were prepared by the Czochralski technique and polycrystalline ($TbAl_2$) samples were produced by arc melting [4]. Single-crystal discs approximately 6 mm diameter and 1 mm thick were spark-machined parallel to low-index planes. Similar-sized polycrystalline specimens were also prepared. The specimens were mounted in resin and polished to a final 0.25 μm finish using successive grades of diamond paste. The specimens were then examined in a Cambridge S4 SEM fitted with a solid state p–n back-scattered detector. After ensuring that the lapped surface was flat, the amount of surface strain was assessed from the quality of the electron channelling pattern.

Following the initial surface characterization the samples were ion-beam planed. The mounted specimen was placed on the work table of an Ion Tech Microworkshop fitted with a B25 slit source. The ion gun was positioned to produce an incident beam angle of 20° with a sample-to-gun distance of 12 cm. Rotation of the sample was used to compensate for variations in the ion density at the sample. This beam–sample configuration allows a large area, approximately 4 cm^2, to be planed under controlled conditions. The specimens were planed for 5, 10 or 20 h using high-purity argon with an applied voltage of 5 kV and current density of 50 μA. Weight-loss measurements showed that the material removal rate was $\cong 0.5\,\mu m\,h^{-1}$. After ion-beam planing the specimens were re-examined in the SEM.

3. Results and discussion

A typical selected-area channelling pattern (SACP) obtained from a mechanically polished surface is illustrated in Fig. 1a. Although a diffuse pattern is discernable there is insufficient detail to unambiguously identify the crystal orientation. The improvement in pattern quality that is produced when the lapping damage is removed is shown in Fig. 1b. The SACP in this figure was obtained from the same sample as shown in Fig. 1a but after ion-beam planing for 10 h. A good-quality channelling pattern is evident and the crystal orientation can be readily determined; in this case the characteristic 1 0 0 pattern of the cubic Laves phase structure of the RAl_2 compounds. The amount and depth of strain that is introduced into a material during lapping depends on many factors, e.g. polishing conditions, sample, etc. If all the surface damage is not removed from the lapped sample, then an inferior channelling pattern will be obtained. For the rare earth compounds investigated in this study it was found that no discernable improvement in pattern quality was

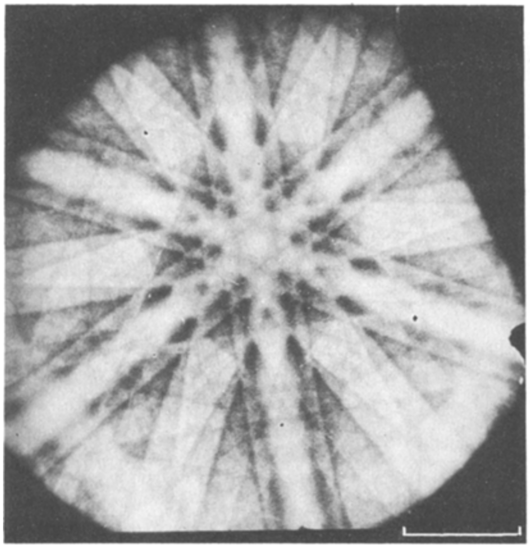

Figure 2 Scanning channelling pattern (30 kV) from a $TbAl_2$ 1 1 1 single crystal after mechanically polishing and ion-beam planing for 10 h. (Marker bar = 1 mm.)

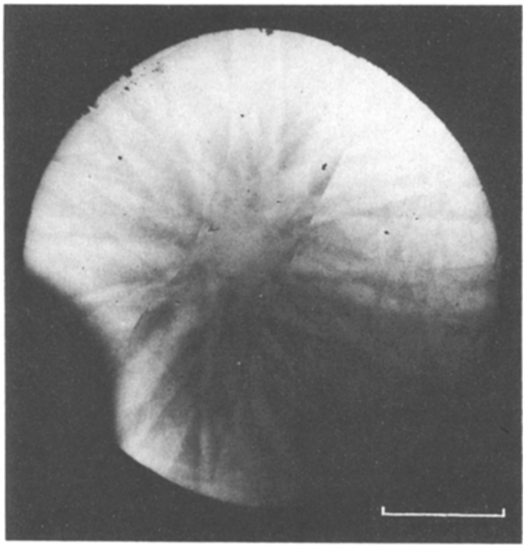

Figure 3 Low-angle boundaries revealed in the scanning channelling pattern (30 kV) from the surface of a $(GdTb)Al_2$ 1 1 0 specimen after mechanically polishing and ion-beam planing for 10 h. (Marker bar = 1 mm.)

obtained for ion-beam planing times in excess of 10 h.

Since it is known that material removal using ion-beams can introduce surface roughening [5], it is important to establish that the procedures described here do not distort the initial surface topography. The flatness of a strain-free surface can be assessed by forming a channelling pattern as the electron beam is scanned over the surface at low magnification, thus generating a scanning channelling pattern (SCP). This is in contrast to the SACP which is generated from a selected area by rocking the beam about a point in the surface. Fig. 2 shows an SCP formed from a sample of $TbAl_2$ which had been ion-beam planed for 10 h. The undistorted and uniform pattern shows that the planing conditions have produced an even removal of material over the whole of the specimen surface (~ 1 cm diameter). Scanning patterns can also be used to identify microstructural features such as low-angle tilt boundaries. The scanning pattern shown in Fig. 3 was obtained from a nominally single-crystal RAl_2 sample. X-ray Laue patterns from this specimen indicated a single crystal and, after ion-planing for 10 h, SACPs from various points on the surface confirmed this deduction. However, the scanning pattern clearly reveals a slightly misoriented region separated from the main lattice by low-angle boundaries.

In addition to showing variations in crystal orientation and lattice perfection, scanning patterns can also be used to highlight grain size and distribution in polycrystalline material [6]. This form of imaging uses the variation in electron channelling with crystal orientation to produce differences in contrast between grains. To prepare a sample for channelling contrast studies the surface strain must be removed without significant differential etching between the grains. Unfortunately, ion-beam planing invariably produces variations in sputtering rates between grains, due to the dependence of the rate on crystal orientation. However, if surface flatness is maintained within the grain then selected-area channelling patterns can still be obtained. A polycrystalline sample of $TbAl_2$ was prepared and ion-planed in the same manner as the single-crystal specimens. Although a small amount of differential etching could be detected, good channelling contrast was obtained as shown in Fig. 4a. The selected-area channelling patterns that were observed from the individual grains, Fig. 4b to d, show that the planing procedure described in this paper can be used to prepare polycrystalline samples for surface characterization. The technique also allows imaging of inclusions relative to grain structure by atomic number contrast; these are evident in this particular sample both within the grains and at grain boundaries

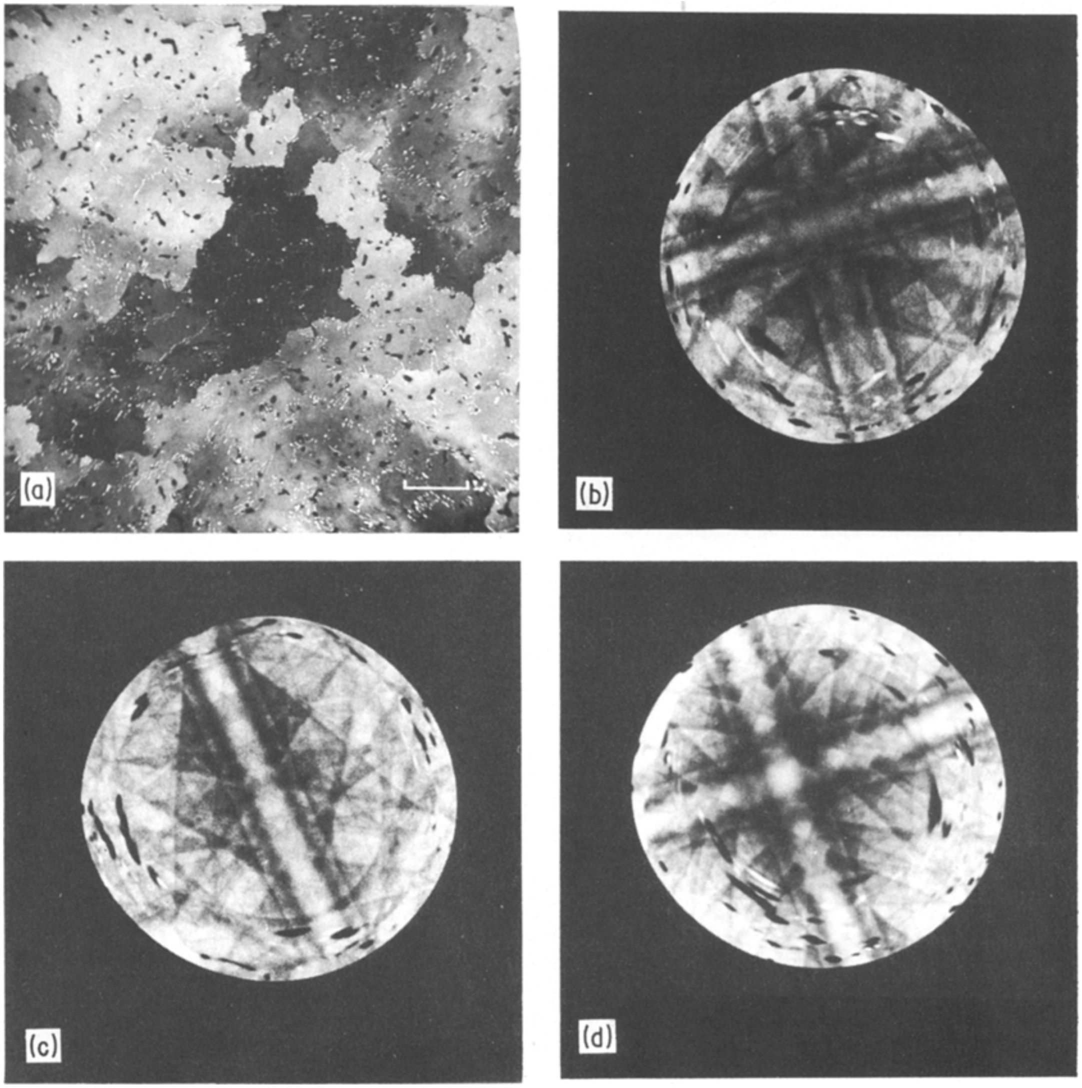

Figure 4 (a) Channelling contrast from a mechanically polished and ion-beam planed $TbAl_2$ polycrystalline specimen together with (b), (c) and (d) SACPs (30 kV) from individual grains. (Marker bar = 100 μm.)

(Fig. 4a) and appear as streaks superimposed on the channelling patterns (Fig. 4b to d).

As well as a preparation technique for the assessment of crystal lattice perfection, ion-beam planing can be used to produce specimens for surface-dependent physical property measurements. For example, the resonance line width that is obtained from $GdAl_2$ specimens during electron spin resonance studies has been observed to depend on the state of the surface. A mechanically polished surface gives a better signal than a spark-machined or chemically etched surface, but further improvement in line width is obtained on removal of the damaged layer from a mechanically polished surface by ion-beam planing [7].

The ion-beam planing procedure described in this paper is not the only method for removing surface-damaged layers; chemical [8] or electropolishing techniques [6] can also be used, and in some cases combined mechanical/chemical etching techniques can produce good results [9]. The main advantages of the ion-beam method is that the same experimental conditions can be used for a wide variety of materials and specimen handling is minimized.

In this paper it has been shown how ion-beam planing can be used to remove surface-damaged layers from the rare earth intermetallic compounds without degradation in the surface quality. Electron channelling patterns from the planed specimens have revealed microstructural detail that was not evident from other characterization measurements. Improved resolution in surface-sensitive physical property measurements has also been obtained.

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