Electron channelling contrast observations in deformed Mg alloys prepared with ion milling

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Abstract. Electron channelling contrast imaging (ECCI) was used in the cold-field emission scanning electron microscope (CFE-SEM) to image the microstructure on deformed bulk specimen. Imaging was conducted with a pole-piece mounted silicon photodiode detector at 5 keV to collect backscattered electrons generated from a low-tilted (0 - 3 degrees) specimen. Broad ion beam milling surface preparation technique was used to remove surface layers and reveal near-surface deformation features. The uniaxial hot-compression tests were conducted on Mg-0.3 wt% Al-0.2 wt% Ca alloy. ECCI observations on deformed bulk specimen showed irregular and complex channelling contrast variations inside parent grains and low angle grain boundaries originated from parent grain boundaries. ECCI on an ion milled prepared surface provides non-destructive and rapid visualisation and characterisation of strain fields along with near-surface deformation substructures in CFE-SEM.

1. Introduction
Understanding hot deformation behaviour of Mg alloys is essential to improve their formability through the activation of multiple slip systems at elevated temperatures. Work hardening, softening and phase transformation processes may occur simultaneously during deformation at high temperatures. The interactions between the three processes may lead to a complex deformed microstructure. The deformed microstructure varies with different deformation variables such as temperature, strain rate and strain [1, 2]. Generally, microstructural evolution during hot deformation involves changes in the shape of grains, increase in grain boundary area and formation of substructures such as low angle grain boundaries inside original grains. At low strains, parent grain boundaries start to bulge out to form serrations. Migration of parent grain boundaries initiates the dynamically recrystallized (DRX) grains formation. At high strain, the microstructure reaches a steady state at which more grain boundaries are covered with DRX grains [1].

1.1. Characterisation technique
Visualisation and semi-quantitative analysis of deformed microstructures have been traditionally performed using colour metallography with polarized light microscopy [3-5]. The advantages of this technique include general microstructure observations at low magnifications, phase identification and semi-quantitative crystallographic data acquisition. The drawbacks of this technique include low...
resolution (~ 20 µm) and low crystallographic sensitivity (misorientation angle > 10°) [6]. Atomic scale microstructure observation and characterisation of deformed materials have been performed using electron diffraction-based techniques in transmission electron microscope (TEM) [7]. However, TEM studies require time consuming and destructive thin foil preparation [8, 9] and may result in non-representative examination area and statistically unreliable results [10, 11]. In addition, deformed materials can be studied using electron channelling contrast imaging (ECCI) in a scanning electron microscope (SEM). SEM studies provide nanoscale spatial resolution, large field of view and statistically reliable information on a bulk specimen [8-10]. Elastic or plastic strain fields can be visualized with ECCI, due to changes in crystal orientation [9]. An elastic strain field causes lateral shifts and rotations in crystal lattice while plastic strain field causes lattice distortion through introduction of dislocations into the crystal. As a result, the intensity of collected backscattered electrons (BSEs) is modulated by the orientation of lattice planes with respect to incident or exit electron trajectories. ECCI micrographs of deformed bulk specimens contain direct evidence of deformation state [12, 13]. Performing ECCI requires appropriate geometry for specimen/microscope, optics conditions and suitable specimen surface [14]. Channelling contrast is commonly detected using either pole piece-mounted Si photodiode detector at low-tilt specimen position or forescatter electron detectors mounted on electron backscattered diffraction camera at high-tilt specimen position [8].

1.2. Surface preparation
For any microstructural observations in a scanning electron microscope (SEM), appropriate specimen preparation is essential such that a mechanical damage free, oxide free, clean and reasonably flat surface is achieved. Suitable specimen surface for ECCI observations can be achieved using chemical or, more commonly, electrochemical polishing techniques after mechanical polishing steps [9]. However, successful electrochemical polishing involves proper selection of variables such as chemical composition and temperature of electrolyte, voltage and time which, sometimes, can be tedious particularly for polycrystalline and multiphase specimens with strained microstructure [13]. Broad ion beam milling is used as an alternative preparation technique to replace electrochemical polishing technique to prepare bulk specimens for ECCI observations [15].

Using appropriate milling conditions for each type of material, an oxide free, clean and reasonably flat surface can be achieved. In addition, rate of material removal from surface can be controlled using the milling parameters. A variety of sputter-induced topographical features have been reported which depend on both specimen conditions and ion beam parameters [16]. A wide range of topographical features was observed in grains with different crystallographic orientations. Such observations include cones, etch pits, bubbles, and periodic and wavelike ripples. Similar artefacts were reported on surfaces prepared with focussed ion beam milling. Formation of all topographical features was attributed to the different localized sputtering yield relative to the surrounding area. For the same ion beam milling parameters (i.e., energy, flow, and angle of incidence), the sputtering yield depends on specimen parameters (i.e., crystallographic orientation, purity, defects and pre-existed topography), and on the surrounding environment (i.e., presence of contamination and neighbouring material). The type and extent of topography development for an arbitrarily chosen specimen is not accurately predicted. Ion beam parameters can be optimized to control the extent of topography development. However, the optimal ion beam milling parameters depend on the specimen conditions and experiments are necessary to determine the best operating conditions for surface preparation [16].

2. Materials and methods

2.1. Casting and heat-treatment
Pure Mg with 99.9 % purity, pure Al with 99.9 % purity and Mg-30 wt% Ca master alloy were used to cast the Mg-0.2 wt% Al-0.3 wt% Ca alloy. Pure Mg was melted at 690 °C in a graphite crucible heated with a high-frequency induction furnace. Pure Al and Mg-30 wt% Ca master alloy were added
to the molten Mg. The melt was held for 15 minutes to ensure Al and master alloy were melted. The melt was cast under SF$_6$ and CO$_2$ gas mixture atmosphere into a preheated copper mould. Homogenisation heat treatment was carried out on as-cast specimens at 500 °C for 8 hours to eliminate the micro-segregation such as precipitates.

2.2. Uniaxial hot-compression tests
Homogenized specimens were machined into cylindrical specimens of 11.4 mm in height and 7.6 mm in diameter. Two ends of the cylinder were covered with a BN paste to minimize the friction between the machine gauges and specimen during deformation. Uniaxial hot-compression tests were carried out using a computer controlled 100 kN servo-hydraulic materials testing system. The tests were performed at 350 °C, strain rate of 0.01 s$^{-1}$ and 0.1, 0.3, 0.6 strains. Specimen was held at deformation temperature for 10 minutes prior to deformation to ensure temperature uniformity throughout the entire specimen. The test was conducted under Ar gas to prevent specimen oxidation. The specimens were quenched immediately after deformation to preserve the dynamically deformed microstructure.

2.3. Specimen preparation
The deformed specimens were cut into halves along the compression axis. Cold mounting was carried out with resin and epoxy. The mounts were ground down to #1200 grit SiC paper. Subsequently, specimens were polished down to 0.05 µm particle size colloidal silica suspension until a scratch-free surface was observed using an optical microscope. In order to remove damaged surface layers caused by the cold-working effects of the grinding and polishing steps, specimen was ion milled at 2 keV ion beam energy and 0.425 s$^{-1}$ specimen rotational speed, using a Hitachi IM-3000 flat ion milling system. For low magnification SE imaging, specimens were ion milled at 0º specimen tilt angle (ion beam perpendicular to specimen surface) for 10 minutes. Ion milling at normal incidence angle induces severe surface topography and is not normally used for bulk surface preparation. For ECCI observations, specimens were ion milled at 85º specimen tilt angle (ion beam almost parallel to specimen surface) for 30 minutes. Finally, prior to insertion into the microscope, a Hitachi ZONEsem sample surface cleaning system was used for 2 hours and 30 % ozone pressure (≈ 227 Torr), to minimize surface contamination.

2.4. Characterisation
A Hitachi SU-8000 FE-SEM was used to study the microstructure after hot-compression tests. ECCI was performed at 0º tilt specimen position (sample surface perpendicular to the electron beam axis), using a photodiode BSE detector inserted below the pole piece (sample surface parallel to the detector). SEM was operated at beam energy of 5 keV and a working distance between 7 - 9 mm for ECCI observations. Acquisition time for each micrograph was 120 seconds. In addition, a SE detector was used with the same parameters. This detector is placed in upper part of the SEM column, and collects SEI and II signals which carry topographical information. All micrographs were obtained from the centre of the specimen to ensure uniform deformation across the examined area. In all micrographs, the compression axis was aligned along the vertical direction. The average grain size of as-received specimen was calculated using the line intercept method [17].

3. Results and discussion

3.1. Deformed microstructure
A uniaxial hot compression test was carried out on polycrystalline bulk Mg-Al-Ca alloy specimen. Figure 1 shows the low magnification SE micrographs of specimens deformed at low and high strains. The specimens were ion milled at 0º specimen tilt angle for SE imaging. Figures 1a and b show the microstructure deformed at 0.1 strain and 0.6 strain, respectively. At 0.1 strain, the microstructure consists of Mg parent grains with internal sub-grain boundaries and extensive serrations at original grain boundaries. At 0.6 strain, a duplex microstructure was observed consisting of elongated parent
grains surrounded with equi-axed DRX grains of 10 ± 3 µm average grain size. Parent grains were elongated perpendicular to the compression axis to accommodate deformation during hot-compression. A light grey grain had a high SE yield and a dark grey grain had a low SE yield. The topographical contrast observed between grains was driven by ion-beam-induced surface topography. For a polycrystalline material, milling rate is a function of local crystallographic orientation [16]. Hence, misoriented regions had different milling rates which consequently, led to different SE yields observed at low magnifications.

Figure 1. Polycrystalline bulk Mg-Al-Ca alloy microstructure after ion milling at 0º specimen tilt angle. Secondary electron micrographs of deformed specimen at (a) 0.1 strain and (b) 0.6 strain. The compression axis was aligned along the vertical direction. The topographical contrast observed between grains was driven by ion-beam-induced surface topography.

For ECCI observations, the specimens were ion milled at 85º specimen tilt angle to remove surface topography and obtain a flat surface. Figure 2 shows low magnification BSE micrographs of specimens deformed at low and high strains. Figure 2a and b show the microstructure deformed at 0.1 strain and 0.6 strain, respectively. In both micrographs, irregular and complex channelling contrast variations were observed across the parent grains. A light grey region was oriented at a non-channelling position and had a high backscattering coefficient. A dark grey region was oriented at a channelling position and had a low backscattering coefficient [12]. In a polycrystalline material subjected to deformation tests, grains deform in a complex and inhomogeneous manner. Each grain is subjected to microscopic constrain from neighbouring grains in addition to the macroscopic applied stress and hence, shows a unique BSE contrast with irregular and regular contrast variations. Type and number of activated slip or twinning systems in individual grains control the microstructure evolution [1].

3.2. Deformation effects in ECCI micrographs
Among Mg parent grains with irregular and complex channelling contrast variations, certain grains had unique channelling contrast in the form of bright bands. Two examples of these grains in specimens deformed at 0.3 strain are shown in figure 3. Figure 3a shows straight bright bands traversing the width of the grains, indicated with black squares. The bands had bright central region surrounded with two lateral dark bands on each side. Figure 3b shows a dark grain containing wavy bright bands with six-fold symmetry originated from a pole, indicated with a black square. Also, the electron backscattered diffraction (EBSD) orientation maps acquired on areas shown in figures 3a
Figure 2. Polycrystalline bulk Mg-Al-Ca alloy microstructure after ion milling at 85° specimen tilt angle. Backscattered electron micrographs of deformed specimen at (a) 0.1 strain and (b) 0.6 strain. The compression axis was aligned along the vertical direction. Irregular and complex channelling contrast variations were observed across the parent grains. and b did not show the same misorientation gradient as was observed in ECCI micrographs. The origin for the differences between the contrast variations in ECCI micrograph and colour variations in EBSD orientation maps is not yet well understood.

Figure 3. Polycrystalline bulk Mg-Al-Ca alloy microstructure after hot-compression test at 0.3 strain. (a) Backscattered electron micrograph of deformed microstructure with bright bands crossing the grains; and (b) backscattered electron micrograph of deformed microstructure containing a grain with wavy bright bands with six-fold symmetry originated from a pole.

In Figure 3b, the shape, contrast and geometry of the observed intersecting bands closely resembled a disturbed electron channelling pattern [12]. Similar channelling contrast observations in the form of irregular dark and bright bands running across the width of the grains were reported on deformed polycrystalline pure Au and Fe-3% Si alloy specimens [12]. Joy et al. [10] used “bend contour” terminology to describe such contrast variations based on the analogy to the bend contours observed on bent foils in TEM. Origin and appearance of observed bend contours in ECCI micrographs were described as follows. At high magnifications, the angular scan of the electron beam is small such that the beam can be considered normal to the surface at all positions. If the grain itself is bent during
deformation tests, the angle between the lattice planes and electron beam changes during scanning the surface from point to point which in turn, changes the BSE intensity [12].

During hot-compression test, a regular change in orientation occurs across the grains due to even bending of the lattice planes. For grains subjected to intense deformation, long-range systematic misorientations in lattice planes result in complex shape and contrast of bend contours. The shape and contrast of bend contours is a function of beam-crystal orientation and incident beam energy and varies from grain to grain due to changes in grain orientation [18]. In a deformed polycrystalline material, grains can be bent about one or multiple axes. If a grain is bent about one axis cylindrically, one single straight band appears in the ECCI micrograph, as shown in figure 3a. If a grain is bent in two dimensions and grain normal is positioned near a low index zone axis, bend contours form a two dimensional zone axis pattern, as shown in figure 3b. In figure 3b, the grain was bent about the zone axis positioned at central pole of the intersecting contours. Hence, bend contours observed on ECCI micrographs can be used to reconstruct the three dimensional concave/convex curvature of the underlying deformed grain. In addition, real-space crystallographic analysis can be performed on bend contours observed in ECCI micrographs since each contour is related to one particular set of diffracting planes [12, 14, 19, 20].

3.3. Appearance of bend contours
In Figures 3b, the appearance of the observed bands has been distorted while the symmetry of the pattern remained unchanged. Plastic deformation causes lattice distortion through introduction of defects such as dislocations into the grains. The presence of dislocations increases the electron beam divergence angle inside the bulk specimen. Increase in divergence degrades the electron channelling pattern quality through decrease in contrast and increase in angular width of the channelling bands. Such quality degradation depends on orientation, local strain conditions and relative deformation temperatures ($T/T_m$), where $T_m$ is the absolute melting point and $T$ is the absolute deformation temperature. Deformation at low relative temperatures ($T/T_m < 0.3$) degrades the pattern quality, however, the pattern geometry remains unaffected and the bands remain straight. Deformation at higher relative temperatures ($T/T_m > 0.3$) distorts the channelling pattern geometry and bands become wavy. This is due to the fact that the orientation being scanned (angular and spatial scan) by the beam varies due to the presence of low angle boundaries [12]. In the present case of $T/T_m = 0.6$, the channelling bands and patterns observed in figures 3a and b were distorted, due to the presence of the low angle grain boundaries. Furthermore, the appearance of bend contours may not be uniform due to the non-uniform bending of the corresponding lattice planes during heterogeneous deformation [21]. For more quantitative analysis on the degree of misorientation inside grains, ECCI micrographs should be compared with the corresponding EBSD orientation maps.

4. Summary
Ion milling surface preparation technique was proved to be beneficial to study deformation of Mg alloys. Milling at low incidence angles revealed low angle grain boundaries and sub-grains with low angle misorientation. Milling at high incidence angle revealed substructures inside grains such as low angle grain boundaries. Furthermore, ECCI observations showed that plastic deformation in Mg grains can be mapped with channelling contrast on an ion milled prepared specimens in FE-SEM. The complicated bend contour patterns observed after ion milling directly maps out the region of severe deformation inside the specimen.

Acknowledgements
The authors would like to thank the General Motors (GM) for their financial support.
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