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# Contribution of texture to the strengthening and fracture in hot-rolled magnesium–12·7 at % cadmium alloy

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The grain size dependencies of the yield and fracture stresses in hot rolled Mg–12.7 at % Cd alloy have been measured in the temperature range 77 to 420 K and are found to be in accordance with Hall–Petch type of equations. In hot rolled Mg–12.7 Cd alloy, the Hall–Petch intercept $\sigma_{0y}$ is higher than that in hot rolled magnesium, while the slope $k_y$ is comparable. The fracture is intercrystalline at 77 K, mixed mode at 300 K and ductile at 420 K. The above flow and fracture behaviours are interpreted in terms of the complimentary effects of texture hardening and solid solution strengthening.

## 1. Introduction

Mechanical processing of materials having hexagonal close packed (h c p) structures produces strong preferred orientations or textures in addition to grain refinement. It is well known that at lower temperatures grain boundaries strengthen metals and the dependence of yield stress, $\sigma_y$, is related to the average grain diameter, $l$, through the Hall–Petch relation [1]:

$$\sigma_y = \sigma_{0y} + k_y l^{-1/2} \tag{1}$$

where $\sigma_{0y}$ and $k_y$ are constants representing the intercept and slope of the Hall–Petch line. In h c p metals eg. Zn, Cd, Mg, Ti, Zr and Be, both these constants are temperature dependent [2–7]. Furthermore, Armstrong [8] has shown that $\sigma_{0y}$ is related to the critical resolved shear stress (CRSS) for the easy slip system operating within the grain volume and $k_y$ is related to the CRSS for more difficult slip/twinning systems required to operate near the grain boundary to maintain continuity of strain across the grain boundary. It is further observed in cadmium materials [9, 10] and in magnesium materials [11, 12], that the Hall–Petch constants are texture dependent inasmuch as the magnitude of the orientation factors relating them to the relevant single crystal resolved shear stresses is decided by the nature and intensity of the texture.

Some of the h c p metals like Zn, Mg and Be exhibit ductile to brittle transition [2, 4, 8] as the temperature is lowered, though the transition is not as sharp as in steels. The brittle fracture stress is grain-size dependent and may be represented by a Hall–Petch type of relation

$$\sigma_F = \sigma_{0F} + k_F l^{-1/2} \tag{2}$$

where $\sigma_{0F}$ and $k_F$ are constants. The influence of grain size and texture on flow and fracture in extruded [4, 11, 14] and hot rolled [15] magnesium has been investigated earlier. These results clearly indicate that $\sigma_{0F}$ as well as $k_F$ are texture dependent.

The aim of the present investigation is to study the contribution of texture to the grain boundary strengthening and fracture behaviour in hot rolled Mg–12.7 at % Cd alloy. The addition of cadmium to magnesium increases [16] the $c/a$ ratio of magnesium and promotes the development of strong basal textures. Cadmium addition also causes considerable solid solution strengthening in magnesium [17].

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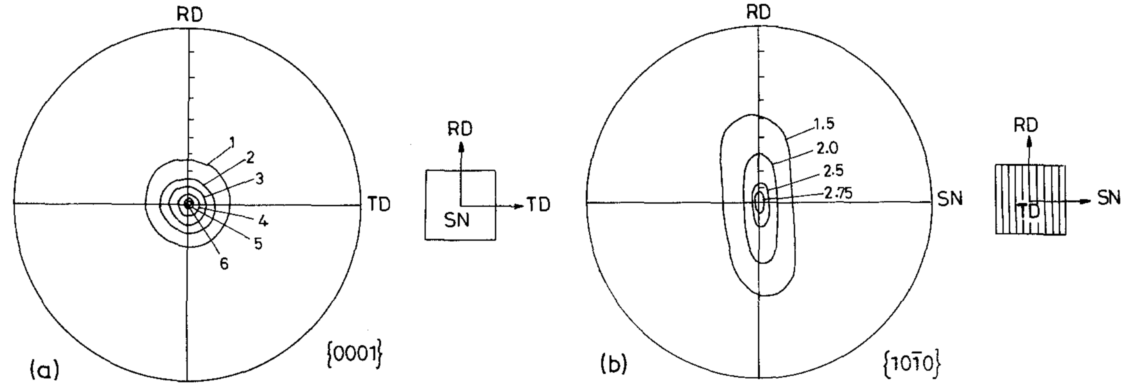


*Figure 1* Pole figures obtained on Mg–12.7 at% Cd sheets rolled at 570 K and annealed at 570 K: (a) {0 0 0 2} reflection on rolling surface, (b) {1 0 $\bar{1}$ 0} reflection on thickness surface. The numbers indicate multiples of random intensity.

## 2. Experimental procedure

Magnesium metal of 99.9% purity and high purity cadmium were used in the preparation of Mg–Cd alloy. Mg–12.7 at% Cd alloy was produced by adding required amounts of cadmium to molten magnesium. Melting operations were carried out in a graphite crucible under a cover of flux consisting of 37.5 wt % KCl, 42.0 wt % $MgCl_2$, 7.5 wt % MgO, 8.5 wt % $CaF_2$, 4.5 wt % BaCl. The alloy was cast in the form of a slab of size 200 mm x 40 mm x 11 mm and chemically analysed after homogenizing at 570 K for 2 h. The assay was close to the nominal composition. The slab was hot forged at 670 K to a final thickness of 8 mm. This was done in four stages each involving a reduction of ≈0.8 mm followed by heating at the forging temperature for 10 min. The thickness of the forged slab was further reduced to 1.6 mm by flat rolling at 570 K. The hot rolling schedule consisted of a reduction of about 0.4 mm per pass followed by heating at 570 K for 5 min. Sheet specimens of 15 mm gauge length and 4 mm gauge width with their tensile axes in the longitudinal direction were milled from the hot rolled sheets. For producing different grain sizes larger than those in hot rolled material, the specimens were annealed in a $SO_2$ atmosphere in the temperature range 570 to 770 K for times ranging from 1 h to 4 h.

The specimens were tested on a tensometer which has a provision for constant cross-head speeds and for cryogenic temperature testing. Tests were carried out at 77 K, 156 K, 300 K and 420 K and at a constant cross-head speed of 0.45 mm $min^{-1}$ (nominal strain rate of $5 \times 10^{-4}$ $sec^{-1}$). For testing at 77 K and 156 K, the specimen, fixed in a cage, was immersed in liquid nitrogen and liquid nitrogen plus alcohol respectively. For testing at 420 K, a thermostatically controlled paraffin oil bath was used.

X-ray pole figures corresponding to (0 0 0 2) and (1 0 $\bar{1}$ 0) reflections were recorded on the sheet surface using the Schulz [18] reflection technique and a Philips texture goniometer. As the pole figures obtained by this technique will cover only the central region with any accuracy (inclination angle 0 to 70°), "composite" specimens with the thickness plane as the reflecting surface were also prepared with a view to obtaining the pole densities in the peripheral region. The "composite" specimens were prepared by slicing the sheet parallel to the longitudinal direction, into pieces with their widths equal to their thicknesses; and bonding them together closely such that the thickness plane becomes the reflecting surface. By combining the pole figure data obtained on the sheet specimen and the "composite" specimen a complete pole figure was constructed.

For optical metallographic examinations, specimens were chemically polished in a solution containing 75% ethyl alcohol and 25% $HNO_3$ and etched in an aqueous solution containing 10% citric acid. The average grain diameter in each specimen was measured using the linear intercept method.

The fracture surfaces of the specimens were viewed in a scanning electron microscope. Care was taken to examine the fracture surfaces immediately after fracture to minimise the problems caused by oxidation.

## 3. Results

### 3.1. Hot rolling textures

The pole figures recorded on the hot rolled Mg–12% Cd alloy sheets are shown in Fig. 1. The {0 0 0 2} pole figure obtained on the sheet surface is given in Fig. 1a while the {1 0 $\bar{1}$ 0} pole figure recorded on the sheet thickness, using the "com-

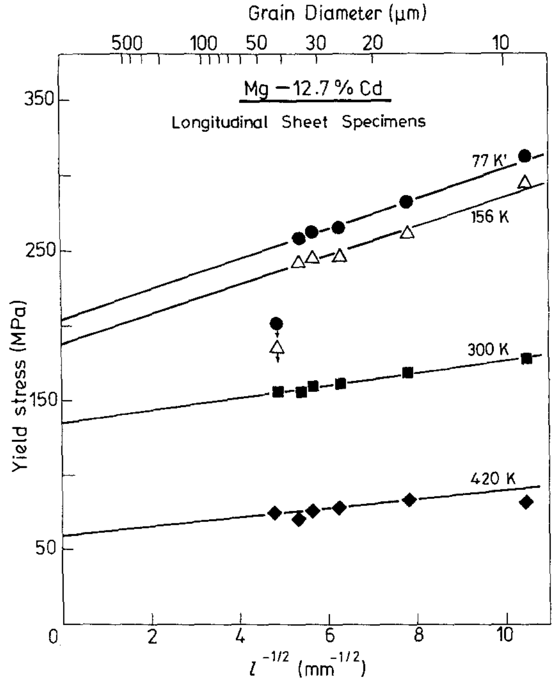


*Figure 2* Variation of yield strength with inverse square root of grain diameter in Mg–12% Cd sheets hot rolled at 570 K.

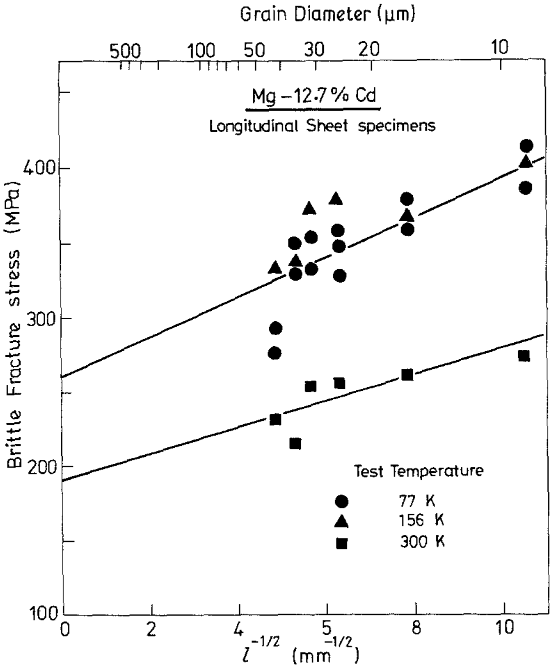


*Figure 3* Variation of brittle fracture strength with inverse square root of grain diameter in Mg–12% Cd sheets hot rolled at 570 K.

posite" specimen is shown in Fig. 1b. The {0 0 0 2} poles are centred around the sheet normal (SN) while the {1 0 $\bar{1}$ 0} poles are centred at the transverse direction (TD).

## 3.2. Tensile properties

The variation of 0.2% yield stress with the inverse square root of grain diameter in the sheet specimen Mg–12.7 Cd alloys obtained at 77 K, 156 K, 300 K and 420 K are shown in Fig. 2. The data obtained in this temperature range obey the Hall–Petch relation (Equation 1). The values of $\sigma_{0y}$ and $k_y$, the Hall–Petch constants, obtained at different test temperatures are given in Table I. The values of $\sigma_{0y}$ and $k_y$ are temperature dependent, being higher at lower temperatures.

The grain size dependence of brittle fracture stress in Mg–12.7 Cd alloys at 77 K, 156 K and 300 K is shown in Fig. 3. The fracture at these temperatures is found to be brittle and the brittle fracture stress, $\sigma_F$, is related to the average grain diameter by a Hall–Petch type of relation (Equation 2). The data obtained at 77 K and 156 K fall on the same line taking into consideration the observed scatter. The data at 300 K fit with a line having a lower intercept and lower

TABLE I Values of Hall–Petch constants $\sigma_{0y}$ and $k_y$ at different temperatures in longitudinal sheet specimens of magnesium and Mg–12.7 Cd alloy

| Specimen | 77 K | | 156 K | | 300 K | | 420 K | |
|---|---|---|---|---|---|---|---|---|
| | $\sigma_{0y}$ (MPa) | $k_y$ (MPa mm$^{1/2}$) | $\sigma_{0y}$ (MPa) | $k_y$ (MPa mm$^{1/2}$) | $\sigma_{0y}$ (MPa) | $k_y$ (MPa mm$^{1/2}$) | $\sigma_{0y}$ (MPa) | $k_y$ (MPa mm$^{1/2}$) |
| Mg–12.7 Cd (hot rolled) | 205 | 9.9 | 188 | 9.6 | 138 | 4.0 | 61 | 2.7 |
| Magnesium (hot rolled) [12] | 123 | 9.4 | 116 | 7.6 | 108 | 5.4 | 40 | 2.5 |
| Magnesium (extruded) [17] | 17 | 15.8 | 12* | 11.5* | 7 | 8.8 | 6† | 2.9† |

*Measurement made at 195 K
†Measurement made at 473 K

TABLE II Values of intercept $\sigma_{0F}$ and slope $k_F$ of the brittle fracture stress against $l^{-1/2}$ line at different temperatures in magnesium and Mg–12.7 Cd alloy

| Specimen | 77 K | | 156 K | | 300 K | |
|---|---|---|---|---|---|---|
| | $\sigma_{0F}$ (MPa) | $k_F$ (MPa mm$^{1/2}$) | $\sigma_{0F}$ (MPa) | $k_F$ (MPa mm$^{1/2}$) | $\sigma_{0F}$ (MPa) | $k_F$ (MPa mm$^{1/2}$) |
| Mg–12.7 Cd (hot rolled) | 260 | 13.2 | 260 | 13.2 | 192 | 8.6 |
| Magnesium (hot rolled) [15] | 180 | 9.4 | 180 | 9.4 | 130 | 9.4 |
| Magnesium (extruded) [17] | 46 | 26.3 | 46* | 26.3* | 46† | 25.0† |

*Measurement made at 195 K
†Measurement made at 473 K

slope than that at 77 K and 156 K. The values of $\sigma_{0F}$ and $k_F$ obtained on Mg–12.7 Cd alloy are shown in Table II.

The SEM fractographs obtained on the fractured surfaces of Mg–12.7 Cd are shown in Fig. 4. At temperatures of 77 K and 156 K the alloy sheets failed in a brittle intercrystalline manner as shown in Fig. 4a. At 300 K, the fracture occurred by a mixed mode (Fig. 4b) consisting of cleavage and dimple features. At 420 K, the alloy failed in a ductile manner as shown in Fig. 4c.

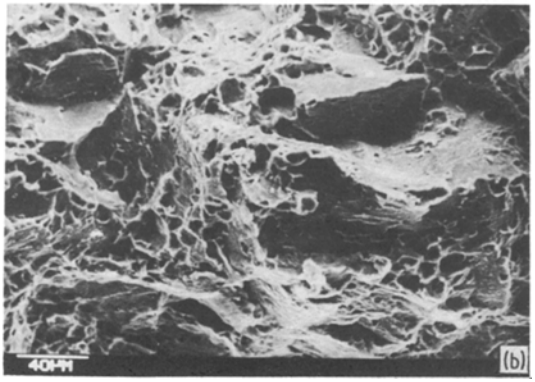
## 4. Discussion

### 4.1. Hot rolling textures

The hot rolling texture in the Mg–12.7 Cd alloy sheets consists of basal planes oriented parallel to the rolling plane. As per the standard projection, the $\{10\bar{1}0\}$ peak at TD suggests that the rolling direction is $\langle\bar{2}110\rangle$. Thus the sheet textures of Mg–12.7 Cd alloy are qualitatively similar to those obtained on hot rolled magnesium [12] and are of the type $(0001)\langle\bar{2}110\rangle$. However, the basal pole intensity of Mg–12.7 Cd is less strong (six times the random) than that recorded on hot rolled magnesium sheets (eleven times the random), which may be attributed to the difference in the deformation processing history. The Mg–Cd alloy is subjected to a lower degree of reduction in hot rolling than magnesium referred above.

### 4.2. Mechanisms of strengthening

In Table I, the values of intercept $\sigma_{0y}$ and the slope $k_y$ obtained for Mg–12.7 Cd, at different



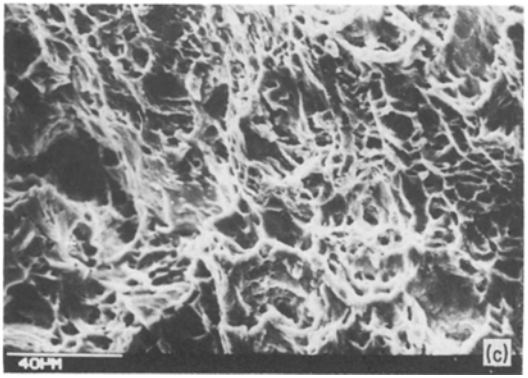


*Figure 4* Scanning electron micrographs of fractures surfaces of hot rolled Mg–12.7 Cd sheets tested in tension at (a) 77 K showing intercrystalline fracture, (b) 300 K showing mixed mode failure, and (c) 420 K showing ductile fracture.

testing temperatures, are compared with those obtained in hot rolled magnesium [12]. Similar data obtained by Hauser *et al.* [4] on extruded magnesium are also included in this table. The results of Hauser *et al.* [4] apply for magnesium with much weaker textures than those obtained in hot rolled magnesium materials. The following characteristics of the alloy are evident from Tables I and II.

(a) The Hall–Petch intercept $\sigma_{0y}$ in Mg–12.7 Cd alloy is much higher than that in magnesium at all testing temperatures, while the $k_y$ values are generally comparable (except at 156 K).

(b) The values of $\sigma_{0F}$ in Mg–12.7 Cd alloy is higher than that in magnesium at all test temperatures.

(c) The $k_F$ values are higher for the alloy at 77 K and 156 K than those recorded for magnesium.

The higher values for $\sigma_{0y}$ in the Mg–12.7 Cd alloy could have been caused by (i) intense textures, (ii) solid solution strengthening and (iii) short range order hardening. These possibilities are discussed below:

The effect of texture on the Hall–Petch constants can be examined once the individual slip systems controlling them are identified. Hall [19] and Petch [20] proposed that the grain size dependence of flow stress is given by Equation 1 could be understood in terms of stress concentration caused by a pile-up of dislocations within the slip bands which propagate slip across the grain boundaries. Armstrong [8] has further shown that the Equation 1 may be expressed as:

$$\sigma_y = m(\tau_0 + k_s l^{-1/2}) \qquad (3)$$

where $m$ is the Taylor orientation factor relating the single crystal shear strength $\tau_0$ to the corresponding polycrystal strength $\sigma_{0y}$, and $k_s$ is the stress concentration required to propagate slip across the grain boundary. On the basis of the pile-up model, Armstrong [8] has further shown that the stress concentration $k_s$ is given by:

$$k_s = C[m^* G\mathbf{b}/2\pi\alpha]^{1/2}\tau_c^{1/2} \qquad (4)$$

where $C$ is a constant, $m^*$ is another orientation factor taking into account the differences in the grain to grain orientations, $G$ is the shear modulus, **b** is the Burgers vector, and $\alpha$ is a constant depending on the screw or edge character of the dislocation pile-up. In h c p polycrystals, Armstrong observed [8] that $\sigma_{0y}$ may be matched with the resolved shear stress $(\tau_0)$ for glide on easiest slip systems while the constant $k_y$ may be matched with the resolved shear stress $(\tau_c)$ for glide on difficult slip/twinning systems operating near the grain boundary, inasmuch as their respective temperature dependencies are similar. The temperature dependence of any shear stress, $\tau$, at a constant strain rate may be expressed as:

$$\tau = B\exp(-\beta T) \qquad (5)$$

where $B$ and $\beta$ are constants and $T$ is the absolute temperature. If $\tau$ consists of a large athermal component, Equation 5 actually gives the temperature dependence of the thermally activated stress in the crystal.

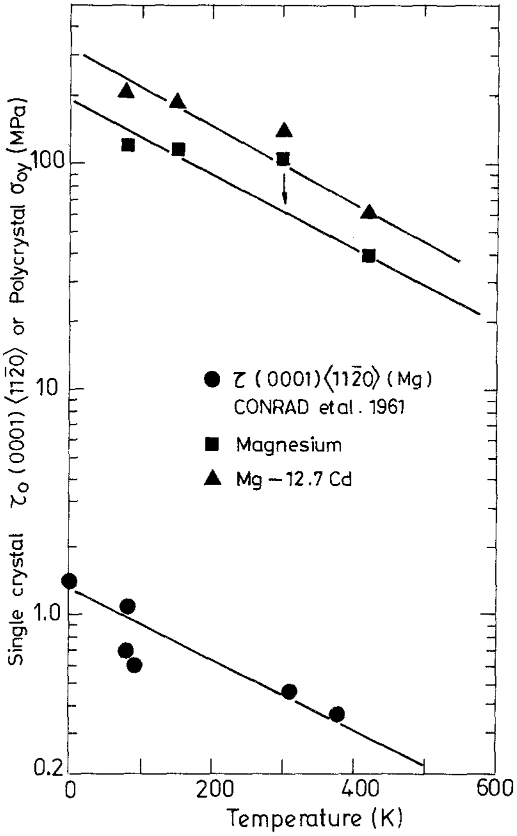


*Figure 5* Temperature dependence of the single crystal resolved shear stress $\tau_0$ for basal slip in Mg and that of $\sigma_{0y}$ for Mg and Mg–12.7 Cd alloy.

The temperature dependence of $\sigma_{0y}$ and $k_y^2$ in Mg–12.7 Cd alloy are shown in Figs. 5 and 6 respectively. As the single crystal data on the temperature dependence of $\tau_0$ and $\tau_c$ for Mg–12.7 Cd alloy are not available, the data for magnesium [21, 22] are used for the purpose of comparison with the temperature dependencies of polycrystal

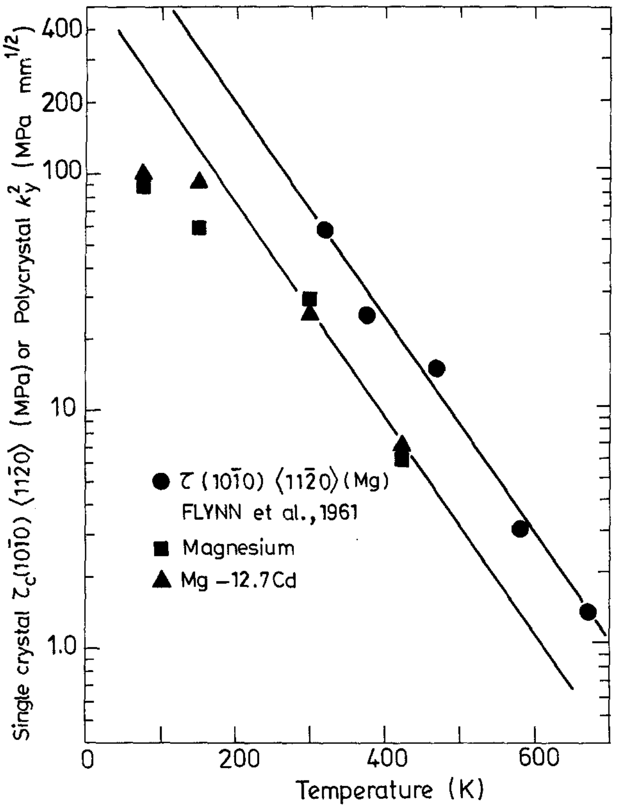


*Figure 6* Temperature dependence of single crystal resolved shear stress $\tau_c$ for prismatic slip in Mg and that of $k_y^2$ for Mg and Mg–12.7 Cd alloy.

(Mg–12.7 Cd) parameters. The reasonable agreement between the single crystal resolved shear stresses and the corresponding polycrystal parameters lends support to the basis that $\sigma_{0y}$ is related to the CRSS for basal slip and $k_y$ is related to the CRSS for prismatic slip, is valid in Mg–12.7 Cd alloy also as in the case of magnesium [8, 12].

From Equations 1 and 3 the relation between $\sigma_{0y}$ and $\tau_0$ may be expressed as:

$$\sigma_{0y} = m\tau_0 \qquad (6)$$

The value of the Taylor orientation factor $m$ is related to the basal texture in the material. When the texture is unfavourable for the occurrence of basal slip, the value of $m$ increases and the material is strengthened. In substitutional solid solution, such as Mg–12.7 Cd, the strengthening due to atomic size difference ($\Delta r$) can be significant and the friction stress $\tau_0$ due to alloying depends on the size factor $\eta$ ($= \Delta r/r$, where $r$ is the atomic radius of the solvent), the atomic concentration ($c$) of the solute, the shear modulus, $G$, through the relation [23]

$$\tau_0 = 1/4 G\eta c \qquad (7)$$

The calculated friction stress ($\tau_0$) for the Mg–12.7 Cd alloy taking $G = 1.75 \times 10^4$ MPa, $\eta = 0.017$ and $c = 0.127$, is $\approx 10$ MPa. In Equation 6, $\sigma_{0y}$ (extrapolated value at absolute zero) is $\approx 300$ MPa (Fig. 5) and with $\tau_0 \approx 10$ MPa, the orientation factor calculates to be about 30. For pure magnesium with weaker textures (Hauser *et al.* [4]) $m = 14$ and the higher value of $m = 30$ in the Mg–12.7 Cd alloy is as expected from the stronger basal textures (6 times the random value). A value of $m \approx 100$ has been estimated for very heavily hot rolled impure magnesium [12] and was interpreted in terms of much stronger textures as well as impurity effects.

In the above discussion, solid solution strengthening due to substitutional solute atoms is only considered. In Mg–Cd alloys, it is well known that considerable ordering also occurs and in the Mg–12.7 Cd alloys, there is a possibility for the occurrence of short range order [24] which can contribute to strengthening [25, 26] to some extent. This has been neglected in the above calculation.

The Hall–Petch slope $k_y$ is related [8] to the CRSS for prismatic slip (Equation 4) and so the intensity of $\{10\bar{1}0\}$ texture will influence the value of the slope $k_y$. A strong basal texture, facilitates the occurrence prismatic slip by orienting prismatic planes in a more favourable position and lowers the $k_y$ values when compared with those in less textured magnesium [4].

### 4.3. Mechanism of tensile fracture

The grain size dependence of brittle fracture stress $\sigma_F$ (Equation 2) is related to $\sigma_{0F}$, the stress required to initiate a microcrack in an otherwise crack free material, and $k_F$, the stress concentration required for the crack propagation. Armstrong [13] observed that brittleness in materials is promoted by large $\sigma_{0y}$ and $k_y$ values. In h c p metals, strong unfavourable basal textures (raising $\sigma_{0y}$ values) and the absence of five independent modes causing plastic incompatibility, promote brittleness. The large increase in $\sigma_{0F}$ (Table II) in Mg–12.7 Cd at low temperatures shows that higher stresses are needed for crack initiation at the grain boundaries compared with that in magnesium. This is essentially due to solid solution strengthening, short range order strengthening and texture strengthening effects. At lower temperatures (<156 K), intercrystalline fracture is observed in Mg–12.7 Cd alloy (Fig. 4a) which is caused by the difficulty of complying with Taylor's criterion of five slip systems, when only basal slip occurs in some grains. Also the occurrence of prismatic slip causes a pile-up of dislocations against the grain boundary and creates a stress exceeding the fracture stress which initiates micro-cracks at the grain boundaries. However, at 300 K, Mg–12.7 Cd alloy sheets failed in a mixed mode consisting of cleavage and dimple features (Fig. 4b). The occurrence of considerable amount of prismatic slip above 300 K provides a preferential transcrystalline path along these slip bands. Also a considerable lowering of melting point of Mg–12.7 Cd (≈ 75 K) helps in increasing the room temperature and elevated temperature ductility and explains the ductile fracture component observed at 300 K and above (Fig. 4b, c).

## 5. Conclusions

From a study of the flow and fracture characteristics of hot rolled Mg–12.7 at % Cd alloy sheets with different grain sizes, the following conclusions could be drawn.

(1) Hot rolling produces strong textures which may be described as near ideal with the basal planes parallel to the sheet surface and the rolling direction along $\langle \bar{2}\,1\,1\,0 \rangle$.

(2) The temperature dependencies of the Hall–Petch constants $\sigma_{0y}$ and $k_y$ are in support of the theory of Armstrong [8] for h c p polycrystals that $\sigma_{0y}$ is related to the CRSS of easy glide system and $k_y$ is related to the CRSS for the difficult glide system.

(3) At all testing temperatures in the temperature range 77 to 420 K the $\sigma_{0y}$ value is higher in the alloy than in magnesium while the $k_y$ values are comparable.

(4) The fracture of Mg–12.7 Cd alloy has been found to be brittle in the temperature range 77 to 300 K while it is ductile at 420 K. The brittle fracture stress is grain size dependent and follows a Hall–Petch type relation.

(5) The mode of fracture is intercrystalline at 77 K and 156 K, while a mixed failure occurred at 300 K in the Mg–12.7 Cd sheets.

(6) The above flow and fracture characteristics are related to the effects of strong basal textures and solid solution strengthening.

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