

# Fatigue of $Mg_3Cd$

## Part 1. *Stress Cycling Behaviour*

R. S. WHITEHEAD, F. W. NOBLE

*Department of Metallurgy and Materials Science, University of Liverpool, Liverpool, UK*

The fatigue behaviour of  $Mg_3Cd$  has been studied under stress-cycling conditions for both single crystal and polycrystalline specimens, in the ordered and disordered state, at room temperature. The results indicate that the polycrystalline material has very poor fatigue resistance for both states of order and that failure probably initiates in basal slip bands. The single crystal results are not directly consistent with earlier work on monotonic deformation, but show fatigue failures at lower stresses than expected.

### 1. Introduction

The effect of superlattice formation on fatigue behaviour has been studied notably in  $Ni_3Mn$ , which forms an  $L1_2$  superlattice, and  $FeCo-V$ , which forms the B2 structure [1]. In the ordered condition deformation by superlattice dislocations causes a marked decrease in cross-slip, leading to homogenous, planar slip, modified cyclic hardening [2] and increased fatigue resistance under stress cycling conditions. No comparable studies have been carried out on hexagonal ordering alloys, and the present work is concerned with one of these,  $Mg_3Cd$ , in which the disordered cph structure may be retained in the quenched condition, while a  $DO_{19}$  superlattice forms on slow cooling [3]. In this material the situation is rather different from that for  $L1_2$  or B2 superlattice formation in that ordering affects the critical resolved shear stresses (CRSSs) of the different slip systems to different extents. Consequently, while the polycrystalline disordered alloy deforms primarily by basal slip, the ordered material deforms equally easily by both basal and  $\{1\bar{1}00\}\langle 11\bar{2}0\rangle$  prismatic slip, leading to a significant change in the deformation characteristics of polycrystalline specimens [5, 6]. Furthermore, the surface slip traces are indicative of coarse, heterogeneous slip – which is more pronounced in the ordered than in the disordered specimens. The present investigation was undertaken, therefore, to attempt to elucidate the effects which these aspects of its unidirectional deformation behaviour would have on the fatigue properties of  $Mg_3Cd$ .

### 2. Experimental Procedure

#### 2.1. Polycrystalline Specimen Preparation

Ingots of  $Mg_3Cd$  were cast from high purity cadmium (99.999%) and magnesium (99.9%) under an argon atmosphere. The material was hot rolled at 250°C and square section specimens having axes parallel to the rolling direction were cut from the resulting slab. The specimens were annealed at 400°C for 30 min to give a grain size of 0.05 mm and after electropolishing in a 10% nital solution the final dimensions of the gauge length were  $8 \times 2 \times 2$  mm.

#### 2.2. Single Crystal Preparation

The preparation of single crystals of  $Mg_3Cd$  has been described previously [7] and the same procedure was followed here. Fatigue specimens were cut from the as-grown single crystals using a jeweller's saw and were ground to shape using very fine emery paper. An electropolished gauge length, 8 mm in length, was prepared using 10% nital solution and the cross-section dimensions were  $1.6 \times 2.5$  mm. The crystals were finally vacuum annealed for 1 h at 400°C and their orientations determined from Laue photographs.

#### 2.3. Ordered and Disordered Specimens

As in the previous work [4] specimens were fully ordered by slow cooling from 180°C while disordered specimens were obtained by quenching from this temperature. As ordering of the initially disordered specimens will occur very slowly at room temperature – and possibly be accelerated in fatigue – tests on disordered crys-

tals were generally completed within 90 min of quenching. No evidence of ordering during these tests was observed in powder patterns taken from fatigued specimens and one or two specimens tested at  $-15^{\circ}\text{C}$  gave results which were consistent with the room temperature data. In addition, the slip morphology of fatigued disordered specimens was typical of that observed in short-term tensile tests and was very different from that of the ordered material.

#### 2.4. Fatigue Testing Procedure

All fatigue tests were performed in pulsating tension-compression. The majority of the tests were carried out using a Goodman vibrator, operating at a frequency of 75 cycles per sec. The stress on the specimens was built up gradually over the first few thousand cycles and the strain was measured using a capacitance gauge attached to the drive-rod of the vibrator. A more detailed description of the machine and testing procedure is given elsewhere [8].

### 3. Experimental Results

#### 3.1. Polycrystalline Specimens

The fatigue behaviour of both ordered and disordered polycrystalline specimens is shown in fig. 1, the results in this stress range being so similar as to be represented by a single line through both sets of points. A limited number of

gest a fatigue limit of  $\sim 3 \text{ kg mm}^{-2}$  for both states of order; below this stress level negligible amounts of slip were visible on the surfaces of the specimens. Two of the results for disordered specimens showing lifetimes in excess of  $5 \times 10^6$  cycles were derived from specimens given an intermediate disordering treatment every two hours.

Away from the immediate vicinity of a propagating crack the observed slip traces agreed with the slip systems reported previously for tensile deformation. The disordered material appeared to deform almost exclusively by basal slip while in the ordered material about equal amounts of basal and non-basal slip were observed, the non-basal slip presumably being mainly  $\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$  slip. Although the orientations of individual grains were not determined, and so unambiguous slip system determinations were not made, the different morphologies of basal and non-basal slip traces were such that they could be fairly easily distinguished, and it was found that the formation of striated slip bands appeared to be exclusively associated with basal slip even in the ordered material. An example of the marked striation formation associated with basal slip in the ordered material is shown in fig. 2.

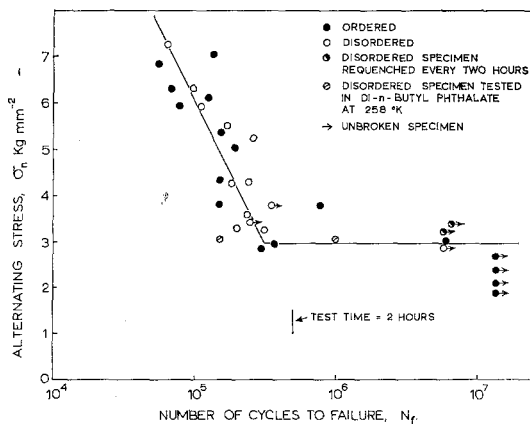


Figure 1 Alternating stress,  $\sigma_n$  vs. No. of cycles to failure,  $N_f$  for polycrystalline  $\text{Mg}_3\text{Cd}$ .

low-frequency, high stress tests did, however, show that the disordered material became increasingly stronger in fatigue than the ordered, at higher stresses [8], reflecting the much higher UTS of the disordered material. The results sug-

Figure 2 Striations on basal slip bands in ordered  $\text{Mg}_3\text{Cd}$  ( $\times 750$ ).

#### 3.2. Single Crystals

The orientations of the crystals required for either basal or  $\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$  prismatic slip were deduced from earlier work [4]. The operative slip systems shown by these crystals in fatigue were verified by two surface analysis. The fatigue behaviour of disordered single crystals of

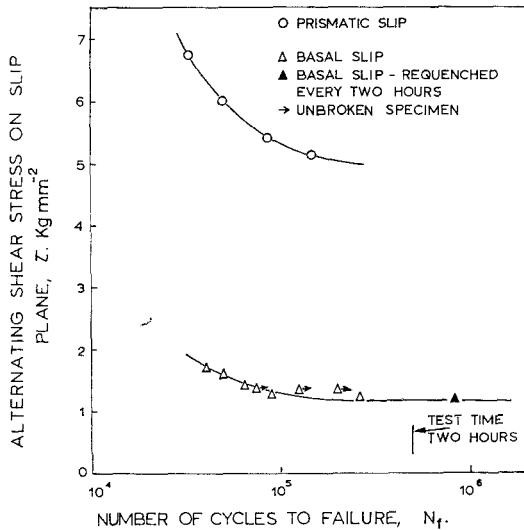


Figure 3 Alternating resolved shear stress vs. No. of cycles to failure for disordered  $Mg_3Cd$  single crystals.

$Mg_3Cd$  oriented for  $\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$  prismatic slip and basal slip is shown in fig. 3. Because of the difficulty of suppressing basal slip in the disordered state, only a limited number of crystals of suitable orientations for prismatic slip were obtained; the results of tests on these are shown in the upper curve. In these crystals, even at the higher stresses used, only a few slip lines not directly associated with the growing crack were observed. In contrast, the disordered crystals oriented for basal slip showed copious slip throughout the entire gauge length even at the lower stresses employed. These observations are consistent with the fact that while the stresses associated with fatigue failure of the basal slip specimens are close to the 0.1% proof stress of the single crystals in tension, the prismatic slip specimens failed at stresses approximately one third of the stress required for appreciable deformation in tension (i.e.  $\sim 5 \text{ kg mm}^{-2}$  compared with  $13 \text{ kg mm}^{-2}$  in tension).

The results for ordered single crystals deforming by basal slip, shown in fig. 4, are characterised by extreme scatter and lack of reproducibility. These crystals exhibited the very coarse slip encountered in monotonic tensile deformation [7], the onset of which in the latter tests was accompanied by a sharp stress drop and was also marked by irreproducibility. The ordered crystals, in addition, showed a much greater tendency for basal cleavage than did disordered crystals. In spite of the scatter, however, the results do indicate that here again the stress levels at which

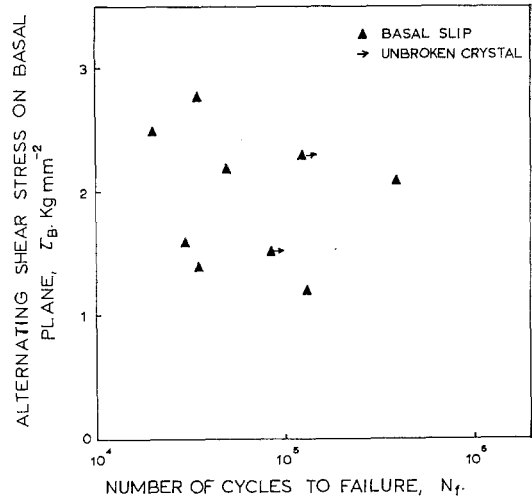


Figure 4 Alternating resolved shear stress vs. No. of cycles to failure for ordered  $Mg_3Cd$  single crystals oriented for basal slip.

both slip and failure were observed in fatigue are considerably lower than the stress required for deformation in tensile tests ( $\sim 5 \text{ kg mm}^{-2}$ ).

Similarly the results shown in fig. 5 indicate that fatigue failure in crystals oriented for prismatic slip, occurs at stresses much lower than the equivalent single crystal 0.1% proof stress in tension ( $\sim 6 \text{ kg mm}^{-2}$ ). However, the results for these crystals were found to be very sensitive to specimen geometry. In particular, when the operative  $\langle 11\bar{2}0 \rangle$  slip vector was within a few degrees of being parallel to a specimen face, much longer lives were obtained. Specimens in this category (orientation A) have been marked with a cross and a curve has been drawn through the remaining points ignoring the "crossed" specimens. With this differentiation of the results at least a lower limit of fatigue behaviour is delineated. This procedure can be justified on the grounds that the single  $\langle 11\bar{2}0 \rangle$  slip vector associated with crack nucleation and growth on a prism plane cannot assist crack growth at  $90^\circ$  to the vector and will, therefore, favour nucleation at a corner, where growth at  $90^\circ$  to the vector is not necessary. When the slip vector makes an appreciable angle with both faces at a corner the direction of crack growth will always be at a reasonable angle to the slip vector and growth should occur relatively easily in such orientations.

#### 4. Discussion

In general, fatigue failures in single crystals have

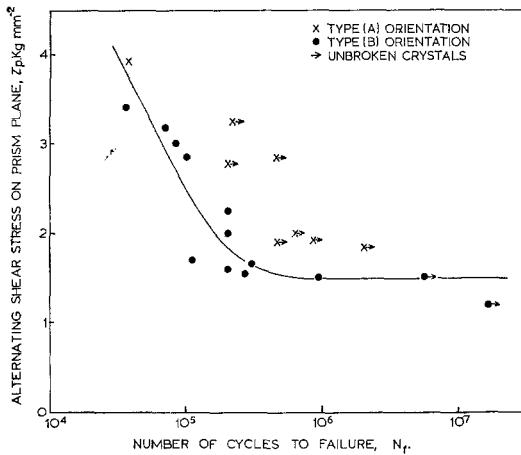


Figure 5 Alternating resolved shear stress vs. No. of cycles to failure for ordered  $Mg_3Cd$  single crystals oriented for prismatic slip.

been reported to occur considerably in excess of the monotonic CRSS and have, for example, been identified with  $\tau_{III}$  in some fcc metals and alloys [9]. A striking aspect of the fatigue behaviour of single crystals of  $Mg_3Cd$ , however, is the small values of the stresses at which slip and failure occur in the present tests compared with the previously reported CRSS (0.1% proof stress) values for similarly orientated crystals. Only for disordered crystals orientated for basal slip are the stresses at which slip and failure occur in fatigue ( $\sim 1 \text{ kg mm}^{-2}$ ) even close to the monotonic CRSS ( $\sim 1.5 \text{ kg mm}^{-2}$ ).

The abnormally low stresses at which failure occurs in single crystals of  $Mg_3Cd$  cannot simply be attributed to pre-existing cracks in the crystals, since slip unassociated with cracking was found at these stresses. If these results are taken at their face value then it is clear that the previous so-called "critical resolved shear stress" derived from tensile tests cannot be taken simply as a measure of the stress required for dislocation movement in these crystals, but must include considerable "pre-yield" dislocation interaction and work hardening.

The present results are, however, reasonably consistent as regards comparability of the single crystal and polycrystalline data, given a suitable choice of orientation factor  $M$  for converting the applied stress on the polycrystals to a shear stress appropriate to the fatigue process. In attempting a similar comparison for single crystal and polycrystalline copper, Kettunen [10] used Taylor's [11] value of 3.06 based on polyslip, and ob-

tained good agreement between the two sets of data. However, in the present material the extent of plastic deformation (as revealed by slip trace observations) was restricted to a fairly small number of grains throughout the gauge length, the remainder of the grains remaining apparently undeformed. It is suggested, therefore, that the appropriate resolving factor should be taken as that for the most favourably oriented grains, since it is in these that the fatigue crack will nucleate. On this basis the resolved shear stress on the slip systems operative in fatigue should be roughly between a third and a half the applied stress, i.e. between 1 and  $1.5 \text{ kg mm}^{-2}$  at the fatigue limit. For disordered  $Mg_3Cd$  this stress range is in good agreement with the apparent fatigue limit of disordered single crystals deforming by basal slip – the operative slip mode in the polycrystalline material. It is, of course, too low for prismatic slip induced failure. For the ordered material the resolved shear stress range is consistent with the stresses at which single crystals oriented for either prismatic or basal slip fail, though metallographic observations, and the geometrical restrictions on failure by prismatic slip, suggest that basal slip induced failure is the appropriate failure mode.

The apparent restriction of the formation of striated slip bands to basal planes, in spite of the availability of equally facile prismatic slip, may well be a manifestation of Nine and Wilsdorf's [12] contention that striation formation is most favoured when parallel slip planes with different operative slip directions are about equally stressed. The three  $\langle 11\bar{2}0 \rangle$  slip directions contained in the basal plane will allow basal slip to conform to this condition: the single slip vector contained by a  $\{1\bar{1}00\}$  prism plane prohibits the attainment of this condition and striation formation by Nine and Wilsdorf's mechanism. The possibility of striation formation in prismatic slip bands must not, however, be discounted completely on this basis since striated slip bands in polycrystalline titanium have been reported [13].

Although the fatigue limit of the polycrystals is consistent with the single crystal data, the fatigue resistance of polycrystalline  $Mg_3Cd$  is extremely poor. The ordered material yields a fatigue limit/UTS ratio of about 0.1; the disordered about 0.07, due to its larger UTS. These values compare very unfavourably with values in the range 0.3 to 0.4 for fcc structures and 0.5 to 0.8 for bcc [14], though on the basis of May

and Honeycombe's work [15], the corresponding ratio for pure magnesium is about 0.15. Low values are not a peculiarity of hexagonal structures, however, since titanium has a value of  $\sim 0.5$  for this ratio [16]. The poor performance of  $Mg_3Cd$  is probably attributable to the fact that the fatigue failures occur at stresses very close to the stress at which slip is first observed, presumably as a consequence of the occurrence of coarse, planar, heterogeneous slip in both states of order – a very poor combination of properties from the point of view of fatigue resistance. It is worth noting that the agreement between single crystal and polycrystalline data, together with metallographic observations suggest that the poor fatigue limit/UTS ratio in polycrystals is not due to grain boundary cracking, the mechanism found to be responsible for a low value of this ratio (0.21) in  $\beta$ -brass [17].

### 5. Conclusions

(i) The low-stress, long-life time fatigue properties of polycrystalline  $Mg_3Cd$  are largely unaffected by degree of order.

(ii) The stresses at which fatigue failures were observed in single crystals of  $Mg_3Cd$  are at variance with previously reported CRSS values for basal and prismatic slip, but are consistent with the fatigue properties of polycrystalline  $Mg_3Cd$ .

### References

1. R. C. BOETTNER, N. S. STOLOFF, and R. G. DAVIS, *Trans. TSM-AIME* **236** (1966) 131.

2. A. J. MCEVILLY and T. G. JOHNSTON, *Internat. J. Fracture Mech.* **3** (1967) 49.
3. A. MOORE and C. V. RAYNOR, *Acta Metallurgica* **5** (1957) 601.
4. J. H. KIRBY and F. W. NOBLE, *Phil. Mag.* **16** (1967) 1009.
5. N. S. STOLOFF and R. G. DAVIS, *Trans. Amer. Soc. Metals* **57** (1964) 247.
6. F. W. NOBLE, J. H. KIRBY, and R. S. WHITEHEAD, *Scripta Met.* **2** (1968) 425.
7. J. H. KIRBY and F. W. NOBLE, *Phil. Mag.* **19** (1969) 161.
8. R. S. WHITEHEAD, Ph.D. Thesis, Univ. of Liverpool (1970).
9. G. RUDOLPH, P. HAASEN, B. L. MORDIKE, and P. NEUMANN, *Proc. 1st Int. Conf. on Fracture* **2** (1965) 501 (Sendai, Japan).
10. P. O. KETTUNEN, *Phil. Mag.* **16** (1967) 253.
11. G. I. TAYLOR, *J. Inst. Metals* **62** (1938) 307.
12. H. D. NINE and D. KUHLMAN-WILSDORF, *Canad. J. Phys.* **45** (1967) 865.
13. C. J. BEEVERS and J. L. ROBINSON, *J. Less Common Metals* **17** (1969) 345.
14. A. FERRO, P. MAZZETTI, and G. MONTALENTI, *Phil. Mag.* **12** (1965) 867.
15. M. J. MAY and R. W. K. HONEYCOMBE, *J. Inst. Metals* **92** (1963-64) 41.
16. N. G. TURNER and W. T. ROBERTS, *Trans. TMS-AIME* **242** (1968) 1223.
17. H. D. WILLIAMS and G. C. SMITH, *Phil. Mag.* **13** (1966) 835.

Received 2 June and accepted 19 July 1970