Communications

Effect of Two-Step Aging on the Precipitate Structure in Magnesium Alloy AZ91

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Lagowski[1] has conducted an extensive study of the age-hardening response of Mg-9 pct Al alloys containing 1 to 3 pct Zn (AZ series) subjected to various aging treatments. He showed that a significant improvement, about 10 pct, in the ultimate and yield strengths of Mg-9 pct Al-1 pct Zn-0.3 pct Mn (AZ91) could be obtained by a two-step or double-aging treatment. A brief but inconclusive electron microscope study was also attempted to elucidate the nature of the structures associated with these improved properties. Attainment of a satisfactory polishing technique was a major problem, as described by Roberts.[2]

The purpose of this investigation was to use electron microscopy to compare the single-aged and double-aged precipitate structures of AZ91, and to determine the matrix-precipitate orientation relationship. It was hoped that the basic information obtained would assist in the further development of the AZ series of alloys for commercial applications.

Clark has studied the aging process in Mg-9 pct Al[3] (A9) and Mg-5 pct Zn.[4] In the Mg-Zn system, the precipitate is coherent with the matrix and oriented so as to block basal slip. However, the maximum solid solubility of Zn in Mg is about 8 pct. Thus, the degree of age hardening is restricted by the limited quantity of precipitate. On the other hand, the maximum solubility of Al in Mg is much greater (about 12.5 pct), but unfortunately, the precipitate is non-coherent, it is neither oriented properly to prevent basal slip nor is the inter-precipitate spacing sufficiently small to cause a precipitation hardening effect. Consequently, the age-hardening response of Mg-Al alloys is small.

An alloy of Mg-9 pct Al-1 pct Zn-0.3 pct Mn was chill cast as a 4.5-in. billet and then extruded into 0.5-in. rod. Samples were solution treated at 425°C for 16 h and then given the following aging treatments.

1) 96 h at 100°C, followed by 192 h at 140°C (double-aged).
2) 192 h at 140°C.
3) 192 h at 180°C.

Using a spark erosion cutter, slices 0.016 in. thick were removed from the aged material and discs 0.125 in. in diameter were cut out and dished prior to polishing. Discs were chemically thinned in an 80 pct orthophosphoric acid-20 pct ethanol solution. Electropolishing was done in a solution of 1 pct perchloric acid in ethanol using the low-temperature polishing technique established by Roberts.[2] The cathode was stainless steel, voltage 30 to 50, and the temperature of the electrolyte was −55°C. During polishing, the discs were held in platinum-tipped tweezers.

The polished foils were examined in a Philips EM300 electron microscope equipped with a goniometer stage and double-tilt holder. Selected area diffraction patterns of the matrix and individual precipitates were photographed and measured. A computerized analysis of electron diffraction patterns developed by Milliken[5] was used to determine the precipitate-matrix orientation relationship. The output data from this program whose use is described elsewhere,[6] also includes in-

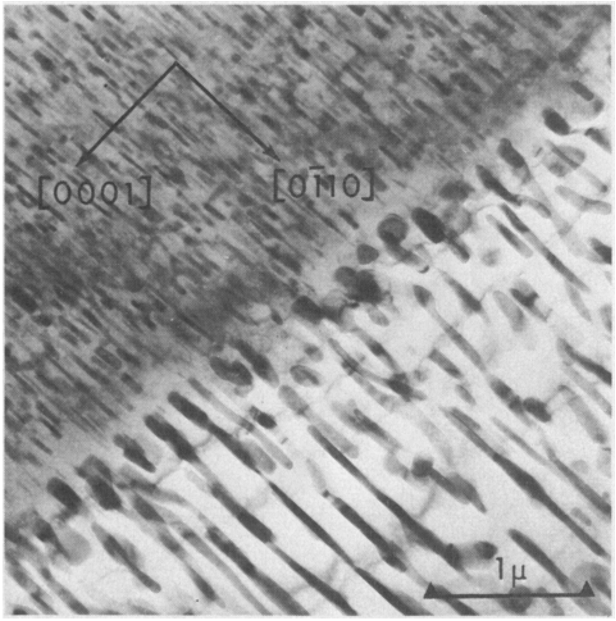

Fig. 1—Electron micrograph of AZ91 after aging for 96 h at 100°C followed by 192 h at 140°C. Photograph shows two regions of precipitation—continuous on the left and discontinuous on the right.

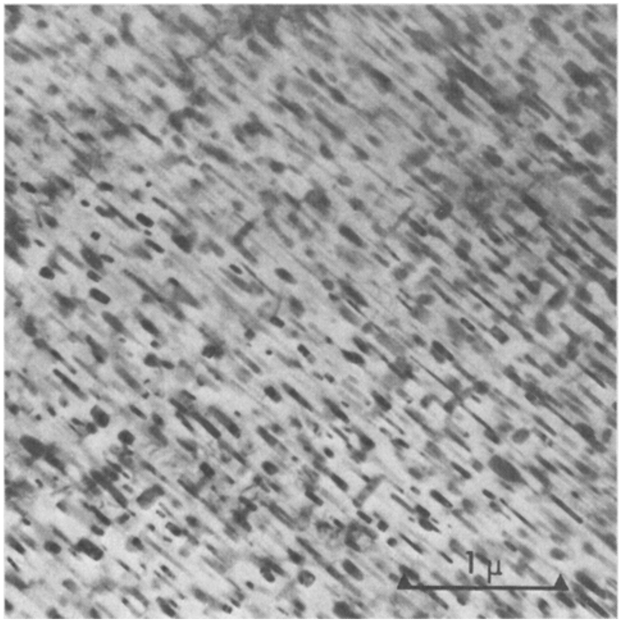

Fig. 2—Electron micrograph of AZ91 after aging for 96 h at 100°C, followed by 192 h at 140°C, showing continuous precipitation.

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formation from which the lattice symmetry and cell dimensions can be determined to within 0.5 pct.

The results of the micrographic examination show that in AZ91, as in A9,[3] two types of precipitates appear. These are:

1) general or continuous precipitates which grow by heterogeneous nucleation; this precipitate is responsible for age hardening; and

2) discontinuous precipitates which nucleate at high angle grain boundaries and grow by a diffusion mechanism; this type of precipitate, massive in form, is detrimental to age hardening of the alloy.

The precipitate in both cases is the bcc $Mg_{17}Al_{12}$. No evidence of a precipitate of another composition was found.

Fig. 1 shows an electron micrograph of a double-aged specimen where regions of two types of precipitation are evident—the platelets of continuous precipitate and the lamellar discontinuous precipitate. The matrix of the discontinuous precipitate appears lighter because of preferential thinning during polishing.

Figs. 2 and 3(*a*) present electron micrographs of general precipitation in a double-aged alloy (Fig. 2) and the alloy aged at 140°C (Fig. 3(*a*)). The distinct difference in precipitate size and distribution are immediately obvious. Subsequent examination of the precipitates showed that two step aging did not result in a change in the type of orientation relationship between precipitate and matrix.

By selected area diffraction, the precipitate-matrix orientation relationships were established. The general precipitate forms as platelets with a basal growth habit. Using the computer program, the precise orientation relationship between the bcc precipitate and the close-packed hexagonal magnesium matrix was determined as

$$[111]_{ppt} \parallel [2\bar{1}\bar{1}0]_{matrix}$$

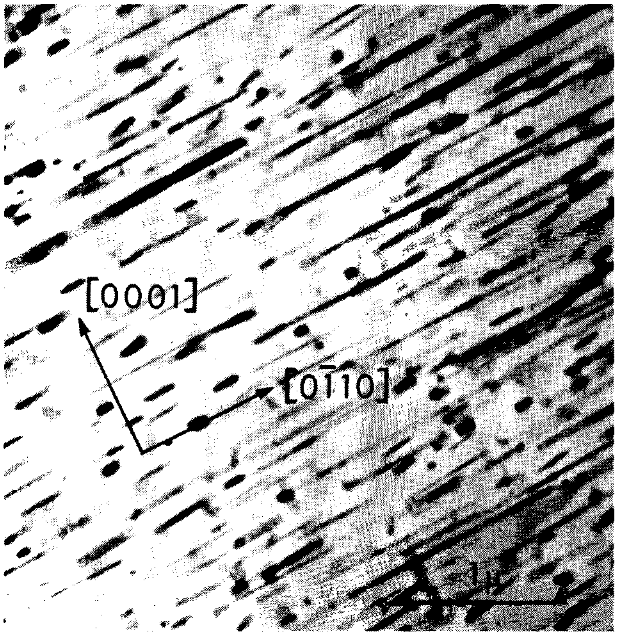
$$(0\bar{1}1)_{ppt} \parallel (0001)_{matrix}$$

Fig. 3(*b*) shows the diffraction patterns of a $[2\bar{1}\bar{1}0]_{matrix}$ and a $[111]_{ppt}$ zone axes. It should be noted that by the threefold substitution of $[\bar{1}2\bar{1}0]$ or $[\bar{1}\bar{1}20]$ in place of $[2\bar{1}\bar{1}0]$ and the two fold substitution of [0001] by $[000\bar{1}]$, six distinct equivalent orientations of the cubic lattice can be generated which obey the above type of relation. Indeed, a single matrix was examined in which three precipitates in three of these orientations were studied.

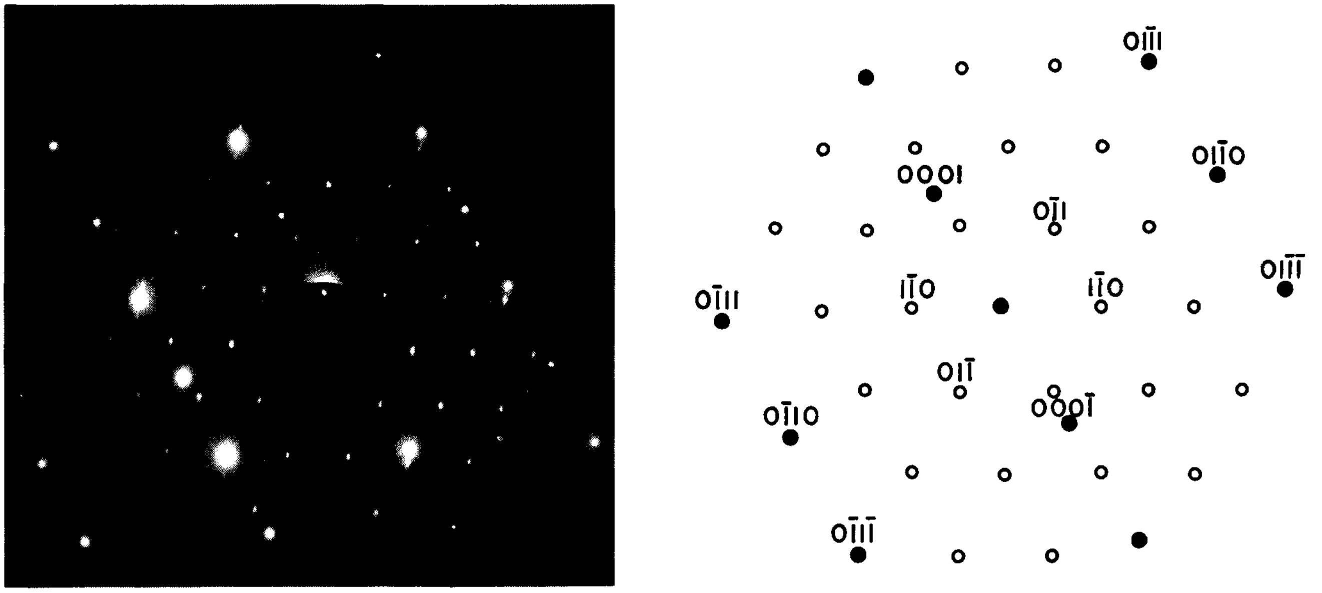
Figs. 2 and 3(*a*) show a few precipitates which lie approximately perpendicular to principal growth habit. By selected area diffraction of one such precipitate, shown in Fig. 4(*a*), the orientation relationship was determined as

$$[2\bar{1}1]_{ppt} \parallel [2\bar{1}\bar{1}0]_{matrix}$$

$$(11\bar{1})_{ppt} \parallel (0001)_{matrix}$$

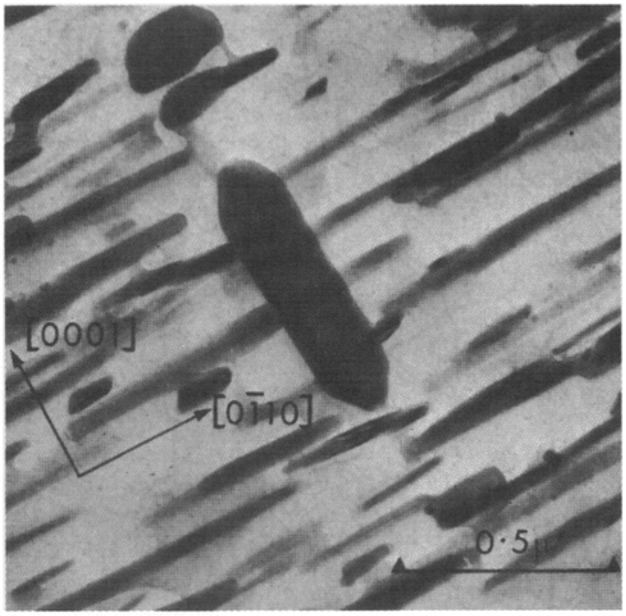


(*a*)

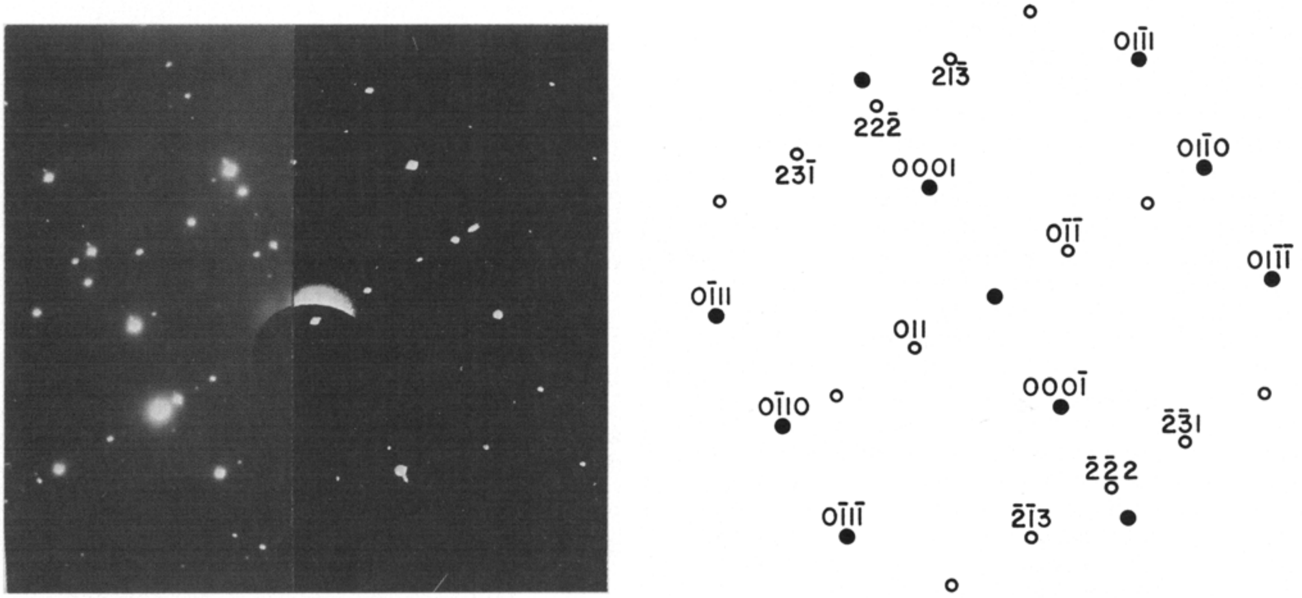


(*b*) (*c*)

Fig. 3—(*a*) Electron micrograph of AZ91 after aging for 192 h at 140°C showing continuous precipitation. (*b*) Diffraction patterns of a $[2\bar{1}\bar{1}0]_{matrix}$ and $[111]_{ppt}$ zone axes. (*c*) Indexed schematic diffraction patterns of (*b*).



(a)



(b) (c)

Fig. 4—(*a*) Electron micrograph showing continuous precipitation in AZ91 after aging for 192 h at 180°C. The large precipitate in center lies approximately perpendicular to the principla growth habit. (*b*) Diffraction patterns of a $[2\bar{1}\bar{1}0]_{\text{matrix}}$ zone axis and a $[2\bar{1}1]_{\text{ppt}}$ zone axis of the large precipitate. (*c*) Indexed schematic diffraction patterns of (*b*).

Examination of symmetry and cell dimensions based on computer output demonstrated that this precipitate was also $Mg_{17}Al_{12}$. The diffraction patterns of a $[2\bar{1}\bar{1}0]_{\text{matrix}}$ and $[2\bar{1}1]_{\text{ppt}}$ zone axes are shown in Fig. 4(*b*) and indexed in Fig. 4(*c*). There are two distinct orientations of the cubic lattice which obey this second type of orientation relationship.

The poor age-hardening response of Mg-9 pct Al was attributed by Clark[3] to two factors:

1) $Mg_{17}Al_{12}$ platelets are not correctly oriented to block basal slip, although some strengthening does occur because $\{10\bar{1}2\}$ twinning is suppressed.

2) the interprecipitate spacing was too large for the precipitate to be sheared by slip dislocations.

Therefore, the only means available to increase the strength of these alloys by precipitation hardening lies in creating a finer dispersion of precipitate. Precipitation in AZ91 after a single step aging treatment is the same as in A9 although Clark[3] did not report a precise orientation relationship, only that the precipitate had a basal growth habit. Thus the expected response of these alloys to variations in thermal aging treatment would be similar.

It is concluded that the increase in strength resulting from a pre-aging treatment reported by Lagowski[1] can be attributed to the decrease in interprecipitate spacing. The orientation of the precipitates was not altered by double aging. Pre-aging serves only to increase the number of nucleation sites for subsequent continuous precipitation.

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