

TECH-NOTES

USING MICROSTRUCTURAL ANALYSIS TO SOLVE PRACTICAL PROBLEMS

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Metallography of Magnesium and its Alloys

Magnesium and its alloys, regardless of the processing procedures employed, are among the most difficult metallic specimens to prepare for microstructural examination. Mg and its alloys are low in hardness and strength but do contain precipitates that are much higher in hardness. This makes it difficult to eliminate scratches and matrix deformation and to control relief, that is, excessive height differences between the matrix and precipitates. Magnesium is also quite reactive and there is considerable debate over whether or not water must be eliminated from the final preparation step or steps. Although its crystal structure is hexagonal close-packed, magnesium does not respond well to polarized light after polishing. The paper presents a new procedure for preparing magnesium and its alloys that yields flat surfaces with no relief problems and minimal scratches and matrix deformation. Some comments about etchants are also made.

Introduction

Preparation of magnesium and its alloys is rather difficult due to the low matrix hardness and the higher hardness of precipitate phases that lead to relief problems, and from the reactivity of the metal. Mechanical twinning may result during cutting, grinding, or handling if pressures are excessive. Final polishing and cleaning operations should avoid or minimize the use of water and a variety of solutions have been proposed. Pure magnesium is attacked more slowly by water while magnesium alloys may exhibit much higher attack rates. Some authors state that water should

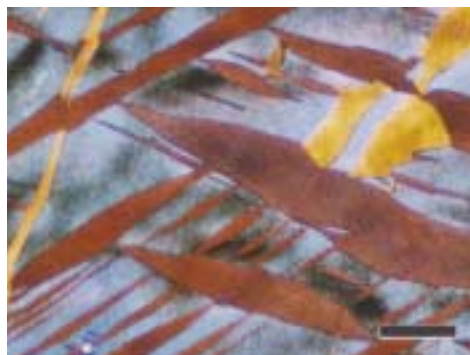


Figure 1. Mechanical twinning observed in wrought, pure (99.98%) magnesium prepared using colloidal silica and etched with acetic-picral (polarized light plus sensitive tint; the magnification bar is 100 μ m long).

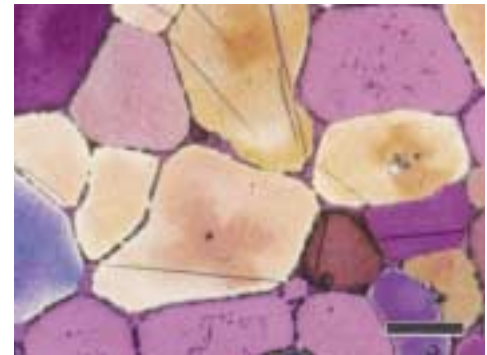
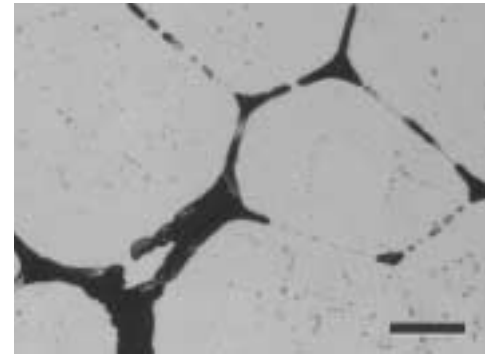


Figure 2a and b. Microstructure of as-cast Mg - 2.5% rare earths - 2.11% Zn - 0.64% Zr revealed by etching with a (top) acetic glycol and b (bottom) with acetic picral (polarized light plus sensitive tint). The magnification bars are 20 μ m and 50 μ m long, respectively. The grain-boundary film of rare earth elements is more easily seen in bright field, and at the higher magnification in (a).

not be used in any step and they use a 1-to-3 mixture of glycerol-to-ethanol or kerosene as the coolant even in the grinding steps. It is always best to grind with a coolant, as fine magnesium dust is a fire hazard. Because of the presence of hard intermetallic phases, relief may be difficult to control, especially if napped cloths are used.

Cutting can introduce considerable damage to specimens and this can be a critical factor in obtaining properly polished specimens. Always use the cutting technique that produces the least amount of damage. An abrasive cut-off saw produces excellent results. Again, a coolant must be used when cutting to minimize damage produced from the heat generated during sectioning. Although magnesium and its alloys have a relatively low melting point, and the solution annealing and aging temperatures are lower, hot compression mounting can be used. However, if the



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specimen were in the as-solution annealed condition, without aging, it would be prudent to use a castable resin, such as epoxy, that generates very little heat during polymerization. The pressure used in hot compression mounting may induce mechanical twinning in high-purity magnesium.

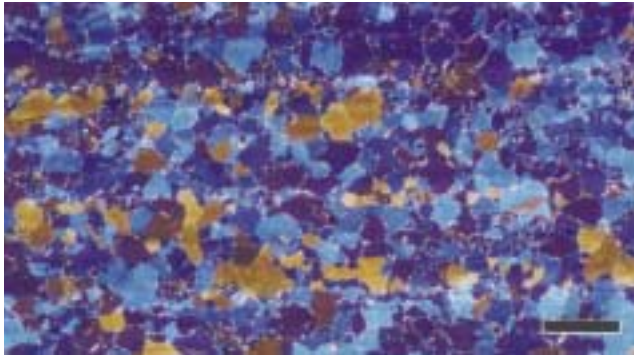


Figure 3. The microstructure of a wrought Mg – 6% Al – 0.92% Zn – 0.3% Mn alloy (longitudinal plane) after etching with acetic picral and viewing with polarized light plus sensitive tint (magnification bar is 100 μ m long).

A number of procedures have been published for mechanical polishing of magnesium and magnesium alloys. Traditional approaches utilize SiC paper to wet grind the specimens using a series of graded abrasive papers from about 240 to 600 grit (P280 to P1200), or even finer. Some metallographers have coated the SiC surfaces with wax to minimize embedment of abrasives. This was tried, and compared to non-coated SiC grinding. Embedding of SiC abrasive particles was not observed. It does not seem to be necessary to coat the SiC paper with wax. Water can be used to flush the grinding debris from the SiC paper surface and minimize heating of the specimen. This is followed by several stages of rough and fine polishing using two or more sizes of diamond abrasive. A coolant/lubricant must be used and these can be water-soluble or alcohol based.

Final polishing has always been a weak point in the process. Magnesium oxide was used but it is difficult to get good quality MgO and the particle size is 1 μ m, which is too coarse. MgO is also hard on polishing cloths. MgO can be suspended in water, but it is best to avoid water. Alumina has been used as a final polishing abrasive, and it is available in sizes down to

Table 1. Five-Step Mechanical Polishing Procedure for Magnesium Alloys

Surface	Abrasive/Size	Load Lb. (N)	Speed rpm/Direction	Time (minutes)
CarbiMet® Waterproof Paper	320-grit SiC (P400) water cooled	5 (22)	250 Comp.	Until Plane
TexMet® 1000 Pad	9 μ m MetaDi® Oil-Based Diamond Slurry	5 (22)	150 Contra	6
TexMet® 1000 Pad	3 μ m MetaDi® Oil-Based Diamond Slurry	5 (22)	120 Contra	5
TexMet® 1000 Pad	1 μ m MetaDi® Oil-Based Diamond Slurry	5 (22)	120 Contra	4
ChemoMet® Cloth	0.05 μ m MasterPolish® or MasterPrep™ Abrasives	3 (13)	120 Contra	1.5-3

* Note: In contra rotation the platen and the specimen holder rotate in opposite directions while in comp. (complementary) rotation, they rotate in the same direction.

Table 2. Chemical Etchants Used in the Study

Name	Composition	Comments
Glycol	1 mL HNO ₃ 24 mL Water 75 mL Ethylene Glycol	General-purpose etch. Immerse for 3-5 seconds; rinse in water and dry.
Acetic Glycol	20 mL Acetic Acid 1 mL HNO ₃ 60 mL Ethylene Glycol 20 mL Water	General-purpose etch. Immerse for 1-3 seconds for cast alloys and up to 10 seconds for solution annealed alloys; rinse and dry.
Acetic-Picral	5 mL Acetic Acid 6 g Picric Acid 10 mL Water 100 mL Ethanol	Immerse until a brown film forms on the surface; rinse and dry. May reveal grain boundaries, mechanical twins and residual cold work.
Phospho-Picral	0.7 mL H ₃ PO ₄ 4-6 g Picric Acid 100 mL Ethanol	Immerse for 10-20 seconds, rinse and dry. Stains the matrix; massive Mg ₁₇ Al ₁₂ phase remains white
Hydrofluoric Acid	10 mL HF 90 mL Water	Immerse specimen for 1-2 seconds. Darkens Mg ₁₇ Al ₁₂ .

about 0.05 μm . It can be purchased as a powder, or as pre-mixed slurries but these are aqueous. Alumina has been used as an aqueous suspension, with additions of soap, and has been mixed with alcohol or ethylene glycol. I have used colloidal silica and this has been effective with pure magnesium but it etches magnesium alloys. Cleaning can be done with water between grinding steps, and after diamond polishing, but generally water is avoided after the final polishing step. Instead, alcohol or other solvents are employed.

Preparation Experiments

A number of experiments were performed in our laboratory to develop a practical mechanical polishing procedure for magnesium and its alloys. A wide variety of specimens, cast and wrought, were tried starting with pure magnesium and then following with popular alloys. First, we tried using one or two SiC steps and then going through a series of diamond (synthetic polycrystalline) polishing steps using MetaDi[®] Supreme Aqueous Suspensions at 9, 3 and 1 μm mean particle sizes. The cloths used were napless, flat pads and several were evaluated. The synthetic chemotextile TexMet[®] 1000 Pad was found to give excellent results. Napped cloths should be avoided as

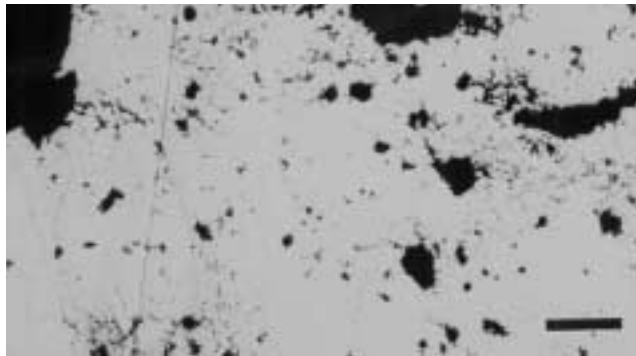


Figure 4. AZ91D surface after the 1 μm diamond step (as-polished) revealing numerous voids (magnification bar is 200 μm long.)

they will produce excessive relief and may lead to other problems, such as drag or pull out. While the aqueous diamond suspensions yielded good results, oil-based suspensions (synthetic monocrystalline diamond) were tried to see if avoiding water would produce better results. In general, no evidence was obtained to suggest that the water in the diamond suspensions attacked the microstructural constituents. How-

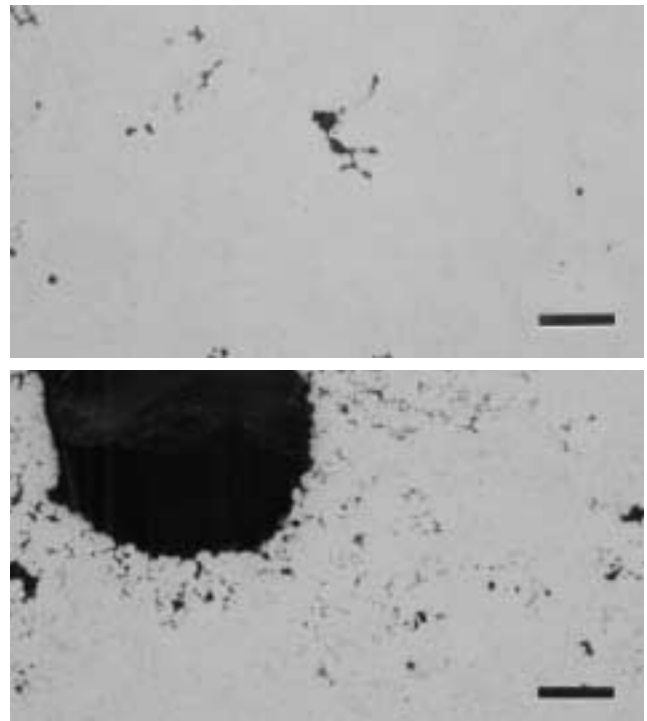


Figure 5a and b. As-polished surface of AM60 (top) and AZ91D (bottom) alloys prepared using MasterPrep[™] Alumina for the final step (magnification bars are 20 μm and 100 μm long, respectively.)

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Shown clockwise from left: **Apex Storage Cabinet** (12" (305mm)), **Apex Hercules H & I**, **Apex Storage Cabinet** (8" - 10" (203-254mm)), **Apex DGD**, **Apex M** and **Apex DGD**.

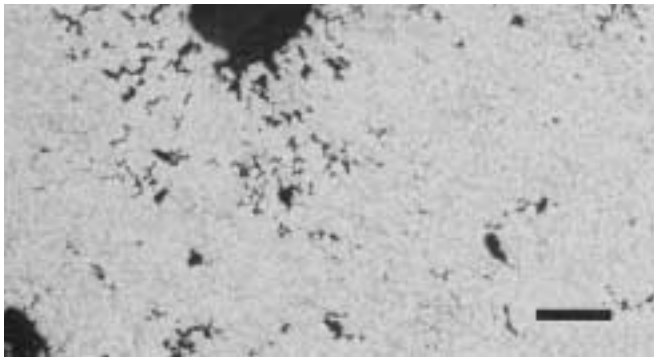
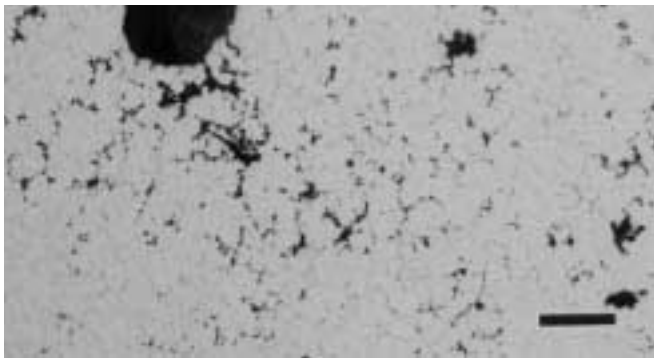


Figure 6a and b. As-polished surface of AM60 (top) and AZ91D (bottom) alloys prepared using MasterPolish® Alumina-silica slurry for the final step (magnification bars are 100µm long).

ever, the greater lubricity of the oil suspension appeared to yield a better surface finish with fewer, shallower scratches.

For final polishing, two cloths were tried. A medium-nap synthetic-suede MicroCloth® Pad and a napless, synthetic polyurethane ChemoMet® Pad were evaluated. It was thought that scratch control might be better with the softer MicroCloth® Pad, while surface flatness (relief control) would be better with the ChemoMet® Pad. However, no relief problems were encountered with either pad, but the results with the ChemoMet® Pad were slightly better. Because the colloidal sili-

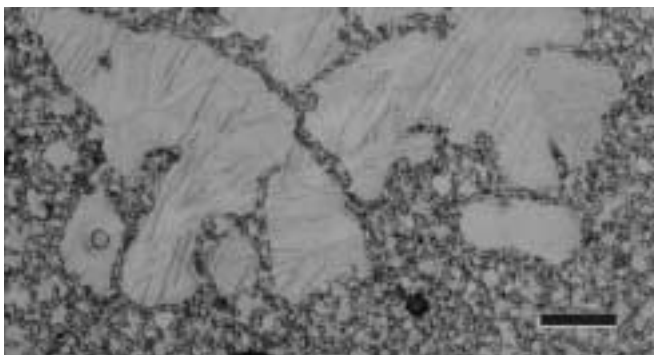
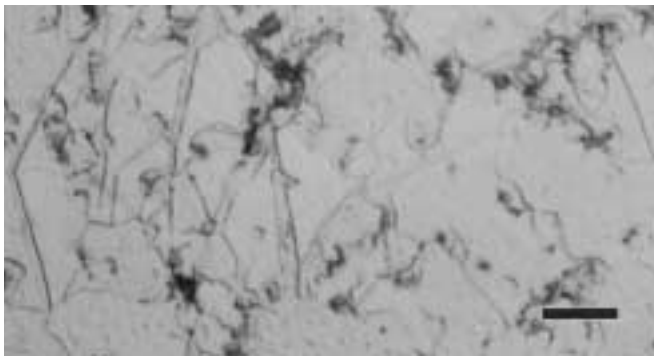


Figure 7a and b. Mechanical twinning observed in AM60 (top) and AZ91D (bottom) alloys prepared using MasterPrep™ Alumina for the final step and etched with acetic-picral (magnification bars are 10µm and 20µm long, respectively).

ca polishing suspension etched the magnesium alloys (but was fine for pure magnesium), other suspensions were tried. Figure 1 shows mechanical twinning in pure wrought magnesium (99.8% Mg) that was final polished with MasterMet® Colloidal Silica. It is not attacked by the high pH of the colloidal silica. Alumina is normally produced by the calcination process, but these abrasives always contain some degree of agglomeration. Recently, alumina made by the sol-gel process has been developed, although only in very fine sizes. MasterPrep™ Alumina Suspension is a sol-gel product with an average particle size of 0.05µm. This abrasive produced satisfactory results. Another proprietary polishing suspension, MasterPolish® Abrasive, was also tried. It is a viscous mixture of alumina and silica and is suspended in a mixture of water, petroleum distillates and propylene glycol. This abrasive also produced satisfactory results. This TECH-NOTE will mainly discuss the preparation and etching of two popular die cast magnesium alloys, AM60 (Mg – 6% Al – 0.15% Mn) and AZ91D (Mg – 9% Al – 0.7% Zn – 0.15% Mn). For thin-walled AZ91D HP (high pressure) die cast parts, both abrasives produced some light etching using a 3 minute polish. No etching was observed on AM60 sections and very light etching was observed on thicker AZ91D sections. The preparation cycle developed from this work is given in Table 1.

The oil-base diamond suspensions must be cleaned from the specimens (and the fixture holding the specimens in an automated process) with alcohol or other suitable solvents. After each diamond-polishing step, the surface was scrubbed with cotton saturated with ethanol. Then, the surface was rinsed quickly with water, sprayed with alcohol, and dried with hot air. Exposure to water was always kept as brief as possible. This cleaning process can be used after the final step, or the specimens can be washed with a glycerol-ethanol solution. Cleaning without using water is inconvenient. Holding the specimen under running water for about a second eased the cleaning problem and did not appear to harm the microstructure. Cosmetic cotton puffs can scratch the surface when swab etching. If desired, a brief vibratory polish with one of the final abrasives could follow the practice. Longer times will produce etching.

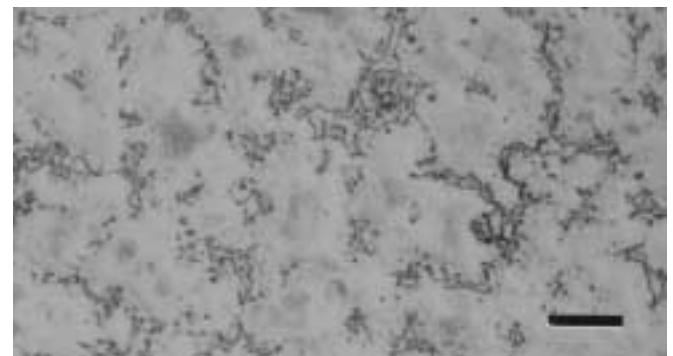
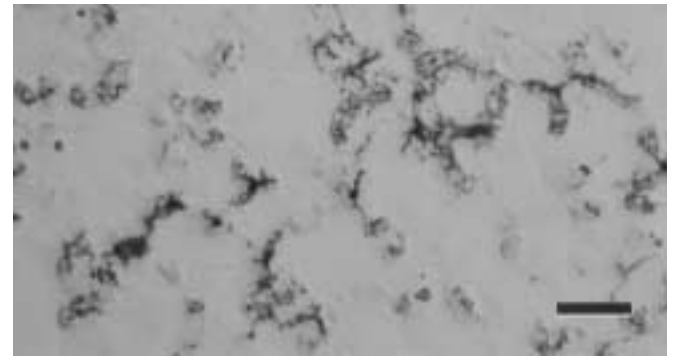


Figure 8a and b. Microstructures of AM60 (top) and AZ91D (bottom) alloys after etching with the glycol reagent (magnification bars are 10µm long).

A variety of standard etchants were used to reveal the microstructure. Table 2 lists these reagents. All are used by immersion. The aqueous hydrofluoric acid solution should be placed in a polyethylene beaker; otherwise glass can be used.

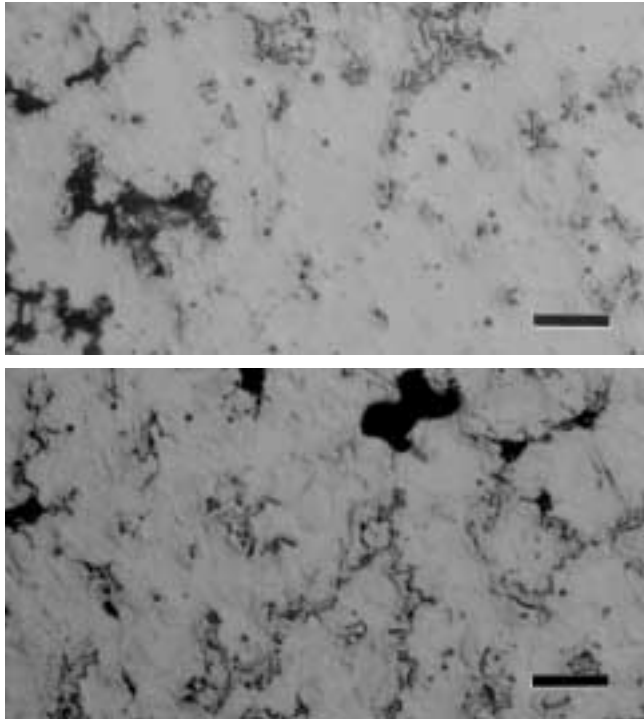


Figure 9a and b. Microstructures of AM60 (top) and AZ91D (bottom) alloys after etching with the acetic-glycol reagent (magnification bars are 10 μ m long).

The glycol etch is preferred for magnesium-rare earth alloys and for magnesium-thorium alloys. Acetic-picral solutions (there are several variations in the literature) are staining reagents and produce color when viewed with polarized light and sensitive tint. The phosphor-picral reagent is preferred when looking for the undissolved second-phase particles in solution-annealed specimens. It produces good contrast between the darkened matrix and the unaffected second-phase constituents. The glycol etchant and the acetic-glycol etchant product similar results, but the later faintly reveals the grain boundaries. The acetic-picral etch also reveals the grain boundaries lightly. These three etchants will outline the massive Mg₁₇Al₁₂ phase. The aqueous HF etch darkens the massive phase preferentially.

Voids may be observed in magnesium alloys and they can come from several sources. Die cast alloys usually exhibit numerous voids due to entrapped air, as will be shown, unless a vacuum die casting approach is used. Cast alloys can exhibit porosity from gas evolution or shrinkage cavities due to insufficient metal feeding during solidification. Eutectic melting may occur during solution annealing. In this case, it is common to see the massive Mg₁₇Al₁₂ constituent around these voids and less elsewhere in the specimen. The massive phase will assume different morphologies depending upon whether or not the alloy contains zinc. If zinc is not present in the alloy, the massive phase forms as a eutectic with the magnesium solid solution. But, when zinc is present, a divorced eutectic is observed with particles of the phase dispersed in the matrix. Further, the massive phase can precipitate in a continuous or a discontinuous manner. During aging above about 200 °C, the massive phase precipitates continuously in a Widmanstätten pattern. Lower aging temperatures and high aluminum contents favor discontinuous precipitation in lamellar form from grain bound-

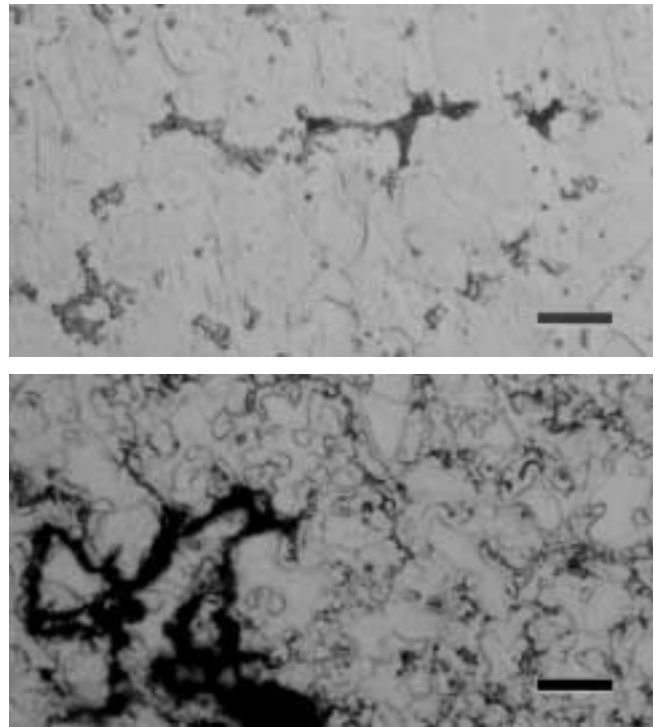


Figure 10a and b. Microstructures of AM60 (top) and AZ91D (bottom) alloys after etching with the acetic-picral reagent (magnification bars are 10 μ m long).

aries. In Mg-Al-Zn alloys, a massive phase can form that is described as Mg₃₂(Al,Zn)₄₉. A 10% aqueous HF solution is reported to blacken the standard massive phase, but not this Zn-motified massive phase. Rare earth (RE) elements have low solubility in magnesium and tend to precipitate at the grain boundaries. Figures 2a and b show the structure of a Mg – 2.5% RE – 2.11% Zn – 0.64% Zr alloy in the as-cast condition etched with the acetic-glycol reagent and with the acetic-picral reagent. Note that the former reveals the grain-boundary RE film better than the latter. Oxide films may also be observed in the matrix. These films formed during solidification and then

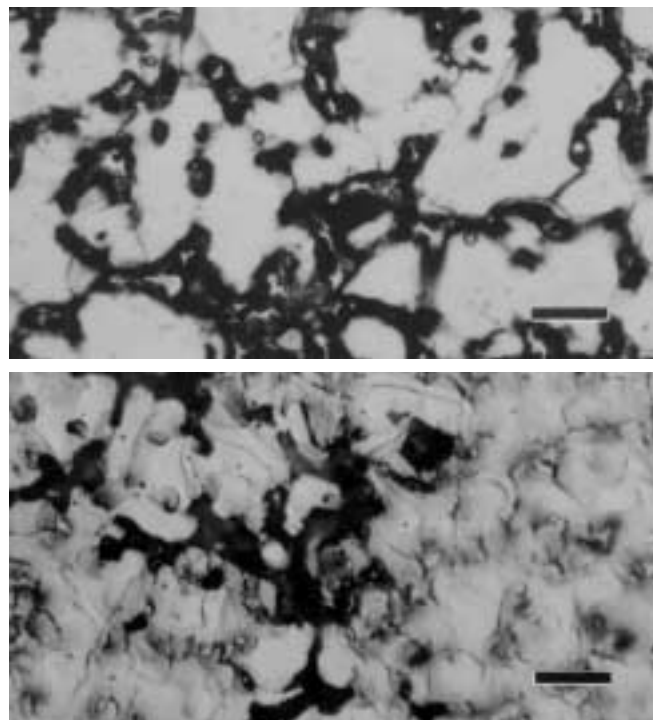


Figure 11a and b. Microstructures of AM60 (top) and AZ91D (bottom) alloys after etching with the phospho-picral reagent (magnification bars are 10 μ m long).

became trapped within the matrix. Wrought alloys are often free of any voids, as shown in Figure 3.

Figure 4 shows the surface of an AZ91D HP die cast specimen after the 1 μ m diamond step. Gas porosity (large round holes) and shrinkage cavities (irregular, narrow holes) are commonly observed in these castings. At the 1 μ m stage, the surface is flat; there are no artifacts from preparation, such as comet tails, pull out, or smeared metal. Contra rotation is more aggressive than complementary and can promote comet tailing or excessive relief at very hard particles. If that is observed, repeat the last step using complementary rotation, and it will be eliminated. Only very fine scratches are present after this step.

Figures 5 and 6 show the as-polished surfaces of AZ91D and AM60 HP die cast specimens after the final step, using MasterPrep™ Alumina and MasterPolish® Suspensions, but before etching. The voids are properly revealed, without any artifacts, and only very fine scratches are visible. A few fine pits aligned in linear fashion can be seen when traversing the surface, but they are infrequent. Figure 7 shows examples of mechanical twins observed in each alloy. The acetic-picral etch was the best for detecting these features. Figures 8 to 12 show the microstructures of both alloys revealed using the five etchants in Table 2. The micrographs clearly show the structures that are always somewhat more complex for the AZ91D with its greater alloy content. With its higher aluminum content, AZ91D exhibits more segregation and contains more of the massive Mg₁₇Al₁₂ phase and more of the very fine precipitated phase than AM60.

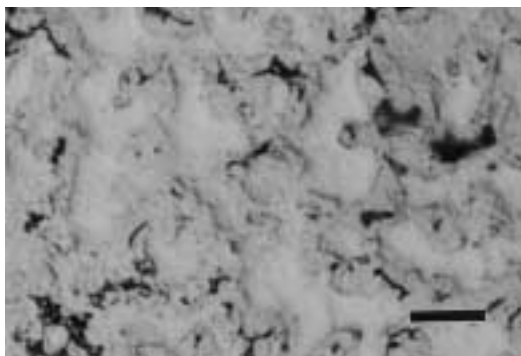
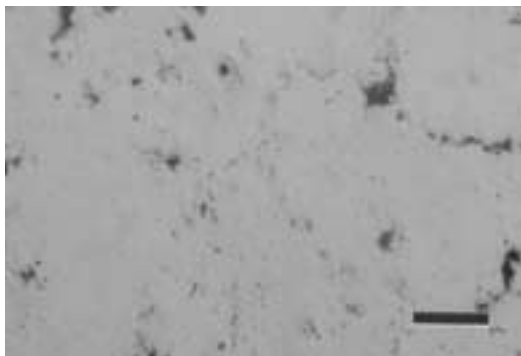


Figure 12a and b. Microstructures of AM60 (top) and AZ91D (bottom) alloys after etching with the 10% HF reagent (magnification bars are 10 μ m long).

Conclusions

Magnesium alloys are difficult to prepare for metallographic examination. All steps must be carefully executed if the end result is to be a true representation of the microstructure. Sectioning must not introduce excessive deformation. Grinding should commence with the finest possible SiC abrasive that will remove the cutting damage in a reasonable time. Oil-based diamond suspensions produced slightly better results than water-based diamond suspensions. Colloidal silica can be used as a final polish for pure magnesium but etches magnesium alloys. MasterPrep™ Sol-gel Alumina and MasterPolish® Suspensions, a mixture of alumina and silica, produced satisfactory final polishing results. The classic etchants do produce somewhat different renderings of the microstructure. Use of only one of the general-purpose etchants (glycol, acetic-glycol or acetic-picral) may be insufficient.

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